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LEAK DETECTION TECHNIQUE IMPROVEMENT STUDY FOR SPACE VEHICLES

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OHIO UNIVERSITY Athens

College of Engineering and Technology

LEAK DETECTION TECHNIQUE IMPROVEMENT STUDY FOR SPACE VEHICLES *

An Investigation and Study

for the

National Aeronautics and Space Administration
George C. Marshall Space Flight Center
Contract NAS8-2563
(NASA

Athens, Ohio
June, 1963

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APPROVED Thomas R. Forbes, Jr.

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OHIO UNIVERSITY
COLLEGE OF ENGINEERING AND TECHNOLOGY
ATHENS, OHIO
DEPARTMENT OF ELECTRICAL ENGINEERING

June 28, 1963

Letter of Transmittal

Mr. Harry A. Street, M-P&C-MEA
Contracting Officer's Representative
Operations Programming Branch
NASA-Marshall
Huntsville, Alabama

Dear Mr. Street:

Attached is the formal report submitted in fulfillment of part 2 d, Article I of NASA Contract Number NAS8-2563. This report covers the period ending May 31, 1963, and covers all work which is in a satisfactory state for reporting.

As directed in Part 2 d, Article I, aforementioned, this report covers a review of the leak detection state-of-the-art (Part 2 a); a discussion of the applicability of available devices and techniques (Part 2 b); a presentation of the results of a search of the existing literature for physical, chemical, or electrical phenomena which might be used advantageously in the detection and measurement of leakages in space vehicle sealing systems.

Following an evaluation of this report by the Vehicle Test Section, Mechanical Systems Analysis Branch, Quality Division, and in accord with their conclusions and direction, additional research and experimental development will be undertaken, if sufficient funds are available, in an attempt to solve those problems pertinent to leak detection. Any technique, method, or system which may improve accuracy, sensitivity, flexibility, automatability, rangeability, ease of application, or reliability will be investigated as extensively as practical and suitable within the limitation of available funds.

Sincerely yours,

R. C. Quisenberry

R. C. Quisenberry, Director
NAS8-2563

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ABSTRACT

A review of the present state-of-the-art in leak detection methods has been made and is reported. Techniques and equipment available have been studied theoretically and experimentally, with respect to shortcomings and/or advantages as applied to Space Vehicle Sealing Systems tests.

An intensive survey of the literature of the past 30 years has been made and an up-to-date bibliography is included. Selected abstracts from these entries are located in Chapter XI.

The salient virtues and shortcomings of the hot anode halogen gun are covered in some detail. Two methods for improving this potentially highly sensitive device have been studied. The differential bridge principle reacts to tracer gradients. The hooded sniffer which flushes the environment with uncontaminated air, has been found of doubtful value.

Among other state-of-the-art devices, tests on two thermistor bridges are described with respect to applicability and limitations.

A miniaturized mass-spectrometer, utilizing the time-of-flight principle, and the Vacion pump, may offer the most sensitive method of detecting leaks, without undue sacrifice of portability.

The detection of leaks by acoustics is described with test results included for the Delcon Ultrasonic Translator. A very interesting

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corollary which seems to offer considerable promise is the sonic injection technique by which sound energy is injected into the system and emerges with the leaking gas. The leak is detected and identified by frequency shift and correlation methods.

Radioactive tracers (alpha emitters) which might trigger resonant cavities into oscillation were not utilized because of their health physics limitations. Organic semiconductors, such as TCNQ, have proven somewhat unsatisfactory due to the rather large concentration of chemical reactant required as a tracer in the leaking gas.

From the systems concept standpoint, microwave information gathering techniques seem most attractive. Using high frequency components in the K or X band, weight and size limitations can be met and information can be obtained in both a space or earth environment.

Conclusions and recommendations are presented as the distillation of the suggestions which have come to light in the course of these investigations. It is hoped that these may result in real progress in the direction of the ultimate leak detector.

AUTHOR

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CHAPTER I

INTRODUCTION

The general problems of leak detection and location are very broad in scope. Much has been accomplished in the field of special problems. Very little of general applicability for shop or field testing of complex systems seems to exist. Much of the very sensitive apparatus loses most of its sensitivity in the face of backgrounds commonly encountered in practical applications. The most sensitive devices are useful in their present forms on vacuum systems but need considerable redesign for pressure application or for work requiring portability.

The study covered by this report has been undertaken under NASA contract NAS8-2563 in an attempt to bring some order out of chaos and to point a way for the possible future development of a leak detection and location system of optimum applicability to space vehicles in the shop, on the pad, and in flight.

A leak can be defined as the result of a hiatus in a system boundary through which an interchange of matter between the system and its environment takes place. Webster (second edition, unabridged) says:

> leak, n. (Prob. fr. ON leki.)
> 1. A crack, crevice, fissure, or hole which (contrary to what is intended) admits water or other fluid, or lets it escape; as a leak in a roof, gas pipe, or ship; figuratively, anything which (contrary to intention) permits admission, or escape or loss, of something; as, a leak in the treasury.

To this might be added remarks about information leaks and various nuclear phenomena arcane to physics.

For purposes of this report it will usually be necessary to supplement the foregoing definition by stipulating certain quantitative considerations. In the interest of consistency all quantitative descriptions of leakages have been reduced to a standard unit of measure - the Standard Cubic Inch per Minute, or SCIM. This means, simply enough, the volume leaking in one minute converted to standard conditions; 68 degrees Fahrenheit and 1 atmosphere. That this is not always an appropriate unit of measure (as in the case of liquids or solids) need not trouble us since this report is concerned exclusively with the problem of gaseous leakage.

Much of the effort expended has been devoted to compiling an up to date bibliography and in abstracting significant papers. This bibliography is included as Chapter X and these abstracts form Chapter XI of this report.

Much simplification has been achieved in the remainder of the

project by limiting the leaks considered to those in which gas is escaping from confining vessels. The art of detecting and locating leaks into vacuum systems is a very advanced and very specialized one, but since such systems do not form a significant part of current missile technology their study has been left to others. Many of the methods of use in detecting such leaks can be applied to outward leaks either with or without modification, and such methods have been included.

The report begins with a study of the general topic of leaks and leak locating from a theoretical standpoint. The phenomena which make leak detection and location possible are reviewed as are those which make it difficult - the noise phenomena, to borrow a term from communication theory. Much of the purpose of Chapter II is to provide perspective for the material which follows. The statement that device A has a sensitivity of 10^{-3} SCIM has little meaning unless something is known about leaks of this magnitude, and the probability of background interference in their detection.

Chapter III deals with devices which seem to merit the designation state-of-the-art with respect to detection and location of leaks of the type with which we are concerned. Information about these has been gleaned from the literature, from advertisements in the trade publications, from replies to direct and indirect inquiries, and from general snooping about in likely places.

In deciding whether to include a device or system in Chapter III, state-of-the-art has been interpreted to mean something like, successful, or will do until the real thing comes along. The old adage that nothing is perfect seems to apply universally to the subject of leak location. The most delicate methods are the least convenient while those which are most neatly and easily applied turn out to be the least sensitive.

One of the main purposes of any state-of-the-art review, of course, is to point out these problems and, hopefully, to suggest possible solutions for them. Thus Chapter III is intended to parade existing useable methods, exhibiting their virtues and their faults. Perhaps a state-of-the-art brigantine and a functional steam engine will inspire Fulton to fruitful results.

Chapter IV discusses in some detail the salient virtues and shortcomings of the hot anode diode halogen gun. The difficulties which beset this device seem mainly due to the weight of the tracer to which it is sensitive. This gives rise to pooling and attendant false indications. Two avenues of approach have been followed in an attempt to improve this potentially highly sensitive device. A closer look at leaks indicates that they are betrayed not only by the presence of the tracer but more significantly by tracer gradients. This suggests the gradient detector which has been investigated and is reported in this

section. A new approach to background contaminants is also tried. This is the hooded sniffer which flushes the environment with fresh uncontaminated air to permit the gathering of meaningful samples from the vicinity of the leak.

As with all such new developments, debugging has been necessary - the course of true invention seldom runs smooth. The differential bridge halogen detector suffers from differences in hot anode diodes. This has lead to the suggestion that a rotary valve be used to time share two sniffer hoses into a single diode. This new device could be interesting from the point of view of information theory and its debugging might lead to an interesting sequence of ideas.

Tests on two thermistor bridges are reported in Chapter V. One of the features noted in both of these leak detectors is that they saturate. Presumably this is inherent in the temperature limitations of the hot element over which the gas must be passed. It should be possible to overcome this limitation by servocontrolling the temperature to hold it constant and using the feedback effort as a measure of the thermal conductivity of the gas being investigated.

The miniaturized mass-spectrometer discussed in Chapter VI is a very challenging topic for consideration. In their currently available forms these instruments are not suitable for the purpose at hand. However, there is so much room for trade-off that it does not seem wise

to sell this instrument short.

When the Delcon Ultrasonic Translator was found to have a transducer sensitive to a narrow band of frequencies in the neighborhood of 40 KC it seemed natural to investigate the philosophy behind this design. A study was proposed and implemented to determine whether anything could be gained by extending this range or by presenting a more complete picture of the acoustical situation. It was thought that the frequency spectrum of the leak being sought might yield some valuable clues.

A study of leaks ranging from nearly zero SCIM to 100 SCIM and from a few psi to 2000 psi and involving ordinary air, nitrogen (dry) and helium revealed that acoustic spectra are fairly flat from 35 KC to 45 KC and fall off reasonably steeply outside the range for all leaks tested. This has been interpreted to mean that the Delcon system is right frequencywise and that improvements must be sought in another department. Chapter VII presents these conclusions and suggests a line of attack now being pursued by the American Gas Association. This is the sonic injection technique by which sound energy is injected into the system to be tested. This energy emerges with the leaking gas and is detected and identified by correlation techniques similar to those which have been perfected for use in radar systems.

Since the original mandate of the contract called for a study of

leaks in a very general sense as they might occur both on the ground and in space, systems concepts have been applied. The question to be answered in this connection is - how can one gather information concerning the state of a system with respect to possible leaks? The answer to this question lies in the selection of a modus operandi of sufficient sophistication and of appropriate informational bandwidth. From this point of view the attractiveness of microwave information collecting techniques becomes obvious. The details of these communications techniques have been so well developed that only the minutiae need worry us. Since high frequency has the advantage of small components, the choice of K band or higher seems suitable.

Only the leak detection method itself need give us much concern - which it indeed does. Two main avenues have been explored. Radioactive tracers (alpha-emitters) which might trigger resonant cavities into oscillation have had to be discarded (though not permanently, it is to be hoped) for reasons of health physics. Organic semi-conductors, of which TCNQ is an example (at present, the only one studied in this laboratory) have proved to be somewhat unsatisfactory. This does not mean that such methods should be written off. New materials are constantly being developed and it is not beyond the realm of possibility that an organic semiconductor more useable in this application will soon be discovered. Chapter VIII

concerns itself with the system concept of leak detection.

Of course any effort at classification of such diverse things as leak detection methods must be either burdensomely overcomplex or end up with miscellaneous leftovers. This report has felt no need to be an exception. Chapter IX notes these loose ends in passing.

One of the main efforts of this study has been the preparation of a complete bibliography on leak detection and location. Chapter X explains and presents this listing.

Chapter XI records some abstracts of papers listed in the bibliography and considered of special interest in the field of leak detection and location.

Conclusions and Recommendations, presented here as Chapter XII, is a distillation of the suggestions which have come to light in the course of these investigations. It is hoped that these can lead to real progress in the direction of the ultimate perfect, weightless, zero cost, infinitely sensitive but not over intrusive leak finder.

CHAPTER II

GENERAL DETECTION AND LOCATION OF LEAKS

General Remarks on the Problem of Leak Detection and Location

A leak from a closed vessel into an environment may be detected by any change in the environment or the vessel caused by or associated with the leak. Changes in any electrical, thermal, acoustical, chemical or other property may be considered to furnish a possible avenue of leak detection and location. The range of such possibilities is indicated by the tabulation below, which is to be considered as representative but not exhaustive:

Property	Sensitive Device	Tracer
Ionization Potential	Halogen Detector	Freon
Thermal Conductivity	Thermistor Bridge	Helium
Dielectric Constant	Vibrating Capacitor	Organic Vapor
Paramagnetism	Inductance Meter	Oxygen
Chemical Composition	Nose; Combustion Meter	Mercaptan; Hydrogen
Atomic Composition	Mass Spectrometer	Helium
Nuclear Stability	Scintillation Counter	Radon
Sound Level	Ear; Microphone	Leak Noise
Temperature	Thermistor	Joule-Thompson Vapors
Electromagnetic	Camera; Radio	Heat; Light, etc.
Color	Eye	Vapor dyes
Pressure	Soap-bubble	None

Range of Required and Attainable Sensitivity

The range of sensitivities of the above tabulated methods is truly enormous. The sonic detectors presently in use, for example, have a lower limit of sensitivity of about 2 SCIM; soap bubble techniques go

down to about 10^{-3} SCIM (lower if a long time is available); thermistor thermal conductivity bridges to 10^{-4} SCIM; halogen detectors to 10^{-6} SCIM; and the mass-spectrometer possibly far below this (to a few molecules per second).

The reasonable sensitivity requirement is also enormously variable. An oxygen leak of pounds per minute might be admissible in a first stage booster about to be fired, while the leak of a toxic mono propellant into the small crew space of a Mars exploration vehicle might reasonably be specified as less than 10^{-6} SCIM, and a leak several orders of magnitude below this (molecules per second) from a satellite might ruin scientific measurements in orbit.

With such a range of sensitivities in mind, it may be said that all leak detection schemes seem appropriate for study.

Factors Involved in the Evaluation of Sensitivity Figures

Figures so far quoted are ultimate sensitivity values. In this study we shall be more immediately interested in realizable, or practical, sensitivity evaluations. Factors which prevent instruments from attaining their ultimate sensitivities may be classified somewhat as follows:

Geometry
Sampling efficiency
Tracer economy
Noise (or contamination)

Geometry enters the picture from the fact that any instrument should and must respond only to local conditions at its sampling inlet(s). Two things are of interest in the leak evaluation process - the space coordinates of the leaking orifice, and the volume or mass rate of leakage. Instruments respond to concentrations of tracer which, in general, may be either matter or energy. The relation between leak coordinates and leak rate, and the concentration of tracer at the instrument is dependent on convection and diffusion.

Convection effects are the more difficult to evaluate. They can be classified as systematic and turbulent. The systematic convection effects depend on the details of the air currents in the vicinity of the leak and are seldom known. Turbulence is a random type of process and therefore closely related to the diffusion process. Turbulence can be thought of as effectively altering (increasing) the diffusion coefficient. The degree of increase of the diffusion coefficient due to the turbulence depends on the degree of turbulence and this is usually not known any better than the characteristics of the systematic currents. Of course, both types of convection are greatly influenced by the geometry of any solid bodies in the neighborhood of the leak.

Diffusion effects in the vicinity of a leak can be calculated when the temperature, pressure, coefficient of diffusion, and local geometry are known. The difficulty here is the computational one of taking into

account the geometry. On the other hand, order of magnitude estimates may be obtained from not unreasonable simplifying assumptions concerning the geometry. For example, at distances large compared to the dimensions (diameters) of the pipes and fittings in the vicinity of the leak, the concentration, C, could be estimated as

$$C = Q/4\pi Dr$$

where Q is the leak rate, D the coefficient of diffusion (in air) of the gas being detected, and r is the distance from the leak. (This is just the expression for a point source diffusing into an isotropic region having no obstructions.) In applying this formula to an actual leak, it must be kept in mind that C can never exceed the concentration of the leaking gas in the system. For this reason r must be several times the reference value r_o at which the concentration equals that in the leaking container

$$r_o = Q/4\pi DC_o$$

At a distance r_o from the leak, the actual concentration could be expected to lie between C_o and about $0.5C_o$ if diffusion is the principal effect. Table I gives the coefficient of diffusion in air for several gases (taken from the American Institute of Physics Handbook and the Handbook of Chemistry and Physics, except where noted), together with r_o values based on a 1 SCIM leak. Fig. 1 gives the relative concentrations as a function of distance from the leak for various gases and for various leak rates. Since the turbulence and

TABLE I

Gas	Temp. °C	D (in air) cm^2/sec	r_o(Q = 1 SCIM) cm
CO_2	0	0.139	0.16
CS_2	20	0.102	0.21
H_2	0	0.634	0.034
O_2	0	0.178	0.12
Argon	20	0.20	0.11
$CC\ell_4$	0	0.064	0.34
He	20	0.632*	0.034
Kr-85	20	0.137*	0.16
Ra-222	20	0.085*	0.25
Freon-12	20	0.071*	0.31

*Calculated from Graham's Law; i.e.,

$$\frac{D_1}{D_2} = \sqrt{\frac{M_2}{M_1}}$$

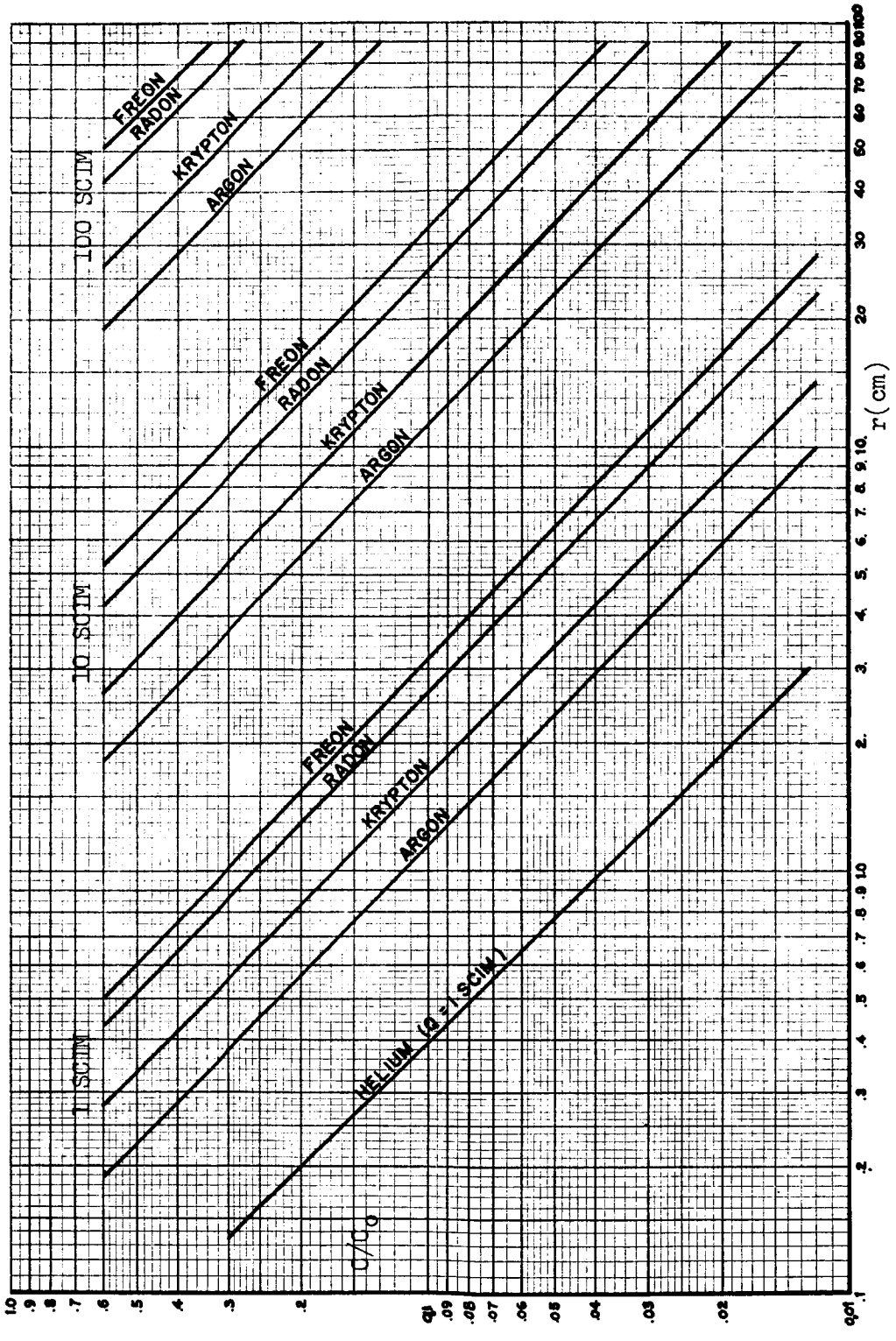

FIGURE 1
CONCENTRATION VS DISTANCE FROM LEAK

geometric effects have been neglected, these graphs are to be considered as useful in a semi-quantitative sense. A feature to be noted is that for a one SCIM leak, the relative concentration of freon-12 would be down to 1% at a distance of 10 inches, due to diffusion alone. Any turbulence would further lower the concentration.

It must be pointed out that the above analysis is probably much oversimplified. In the cases of the hydrogen and helium where the diffusion rate is relatively rapid, steady state may be approached with sufficient rapidity to make these figures somewhat significant, especially when appropriately discounted for the effects of turbulence. With the heavier more slowly diffusing gases, however, the system may never approach steady state because of the effects of turbulence and convection.

In Chapter XII of this report an analysis of diffusion times for freon into closed spaces points up this long time constant difficulty.

One concludes from this analysis that figures for leakant tracer concentrations based on these simplifications must be treated with circumspection and regarded, in any case, as giving at best only upper limits.

Sampling Efficiency

Sampling efficiency may be thought of as measure of how nearly all of the quantity to be measured is used in making the measurement, and of how well extraneous responses can be excluded. Many instruments

must operate with their active parts in a partial vacuum which limits the rate at which samples of the surrounding air can be ingested for analysis. Other instruments may, on the other hand, take in samples so violently that extra turbulences are created in the neighborhood. The sampling problem is somewhat interrelated with the noise and contamination problem.

Tracer Economy

The ultimate sensitivity of most devices on the market is quoted on the basis of 100% tracer concentration in the system or, equivalently, on the amount of tracer leaking. In a practical situation this concentration is necessarily kept down for reasons of safety, economy, sometimes corrosiveness of the tracer. With 1% tracer, of course, the sensitivity figure must be correspondingly altered.

The Problem of Noise and Contamination

Those systems using sonic or electromagnetic energy introduced into the test vessel and detected at the leak have at their disposal the enormously powerful discrimination techniques of the communications sciences to distinguish signal from background noise. Frequency and amplitude modulation, pulse width, precise frequency, phase, etc. may be used to tag the signal.

Gas tracers, on the other hand, whatever the distinguishing feature - radioactivity, ionizability, chemical activity, atomic weight -

are self-obscuring in the presence of random variations in the ambient which disturb their predicted concentration patterns. The problem of distinguishing signal from increasing and randomly varying background contamination may reduce instrument sensitivities by orders of magnitude, or even remove them altogether.

Any gas tracer system, no matter how sensitive, which responds to a simple absolute level of concentration, will soon become incapable of detecting leaks when the ambient tracer concentration rises to the level capable of giving spurious signals. This is the major failing of the simple halogen leak detector, and would also be true of a simple mass spectrometer.

Two solutions to the background problem immediately present themselves: 1) keep the ambient concentration low, 2) utilize a gradient sensor, which detects not the absolute tracer concentration, but the difference in concentrations between two nearby points. This difference would presumably be greater near leaks than elsewhere. Two devices based on application of this principle, the differential thermistor bridge, sensing differences in thermal conductivity, and the halogen bridge, sensing differences in halogen concentration, are described in Chapter IV and Chapter V of this report.

Some Problems of Gradient Sensing

The general pattern of concentration of tracer in the vicinity of leaks is illustrated in Fig. 2. The ratio of the gradient in the ambient streamwise direction, dC/dx to the gradient normal to the ambient flow, dC/dy, will, of course, be proportional to the rate of ambient velocity to the diffusion coefficient of the tracer gas. For example, with equal ambient velocity, halogen tracer will show a long narrow concentration surface, while with helium as the tracer the concentration surface will be broader by a factor of about 9, the ratio of diffusion coefficients.

A rough estimate of the lateral concentration gradient may be made by noting that the time required for the end of a foot long blind duct to reach 10% of the free end concentration of freon is about 6 minutes. The normal convection velocity in the atmosphere is on the order of about 100 feet in 6 minutes. Thus the concentration pattern for freon would be expected to be quite long and narrow.

Given a space distribution of tracer concentration of the type shown in Fig. 2 or Fig. 3, the signal to be expected from a gradient detector will depend on the position of the sampling probes and the response characteristic of the sensing elements. If the response characteristics are linear, as shown in Fig. 4, then the greatest signal will be given by a high concentration of tracer gas and a large separation of the probes. But if the sensor response is nonlinear,

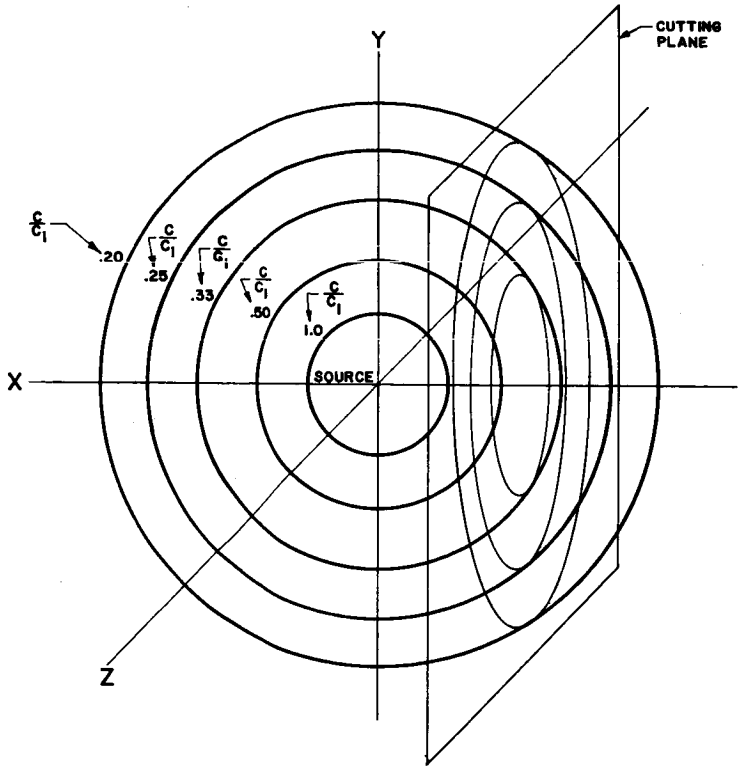

FIGURE 2

ISOCONCENTRATION SURFACES

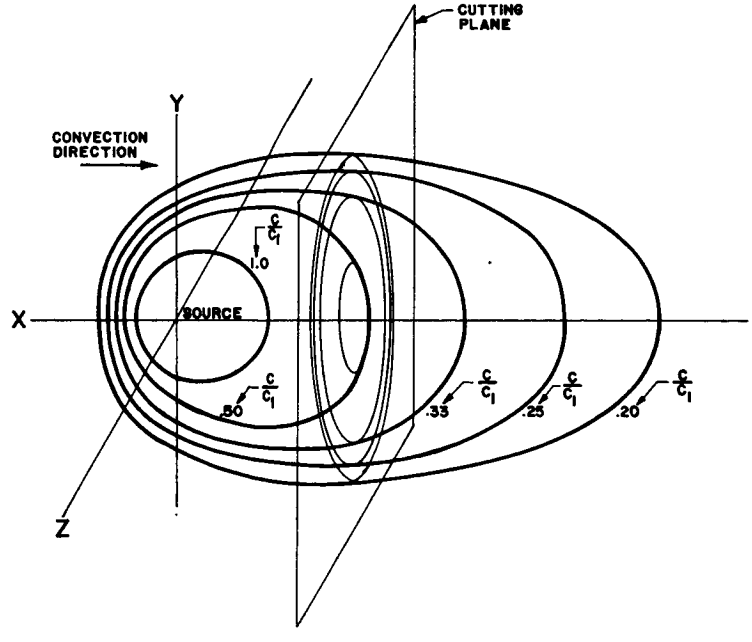

FIGURE 3

ISOCONCENTRATION SURFACES WITH CONVECTION

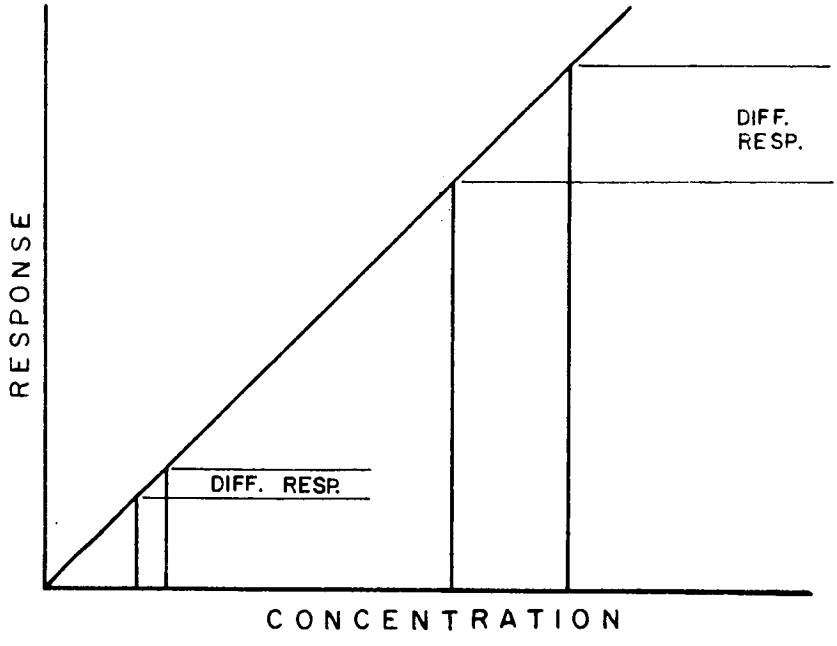

FIGURE 4

DIFFERENTIAL RESPONSE OF LINEAR DEVICE

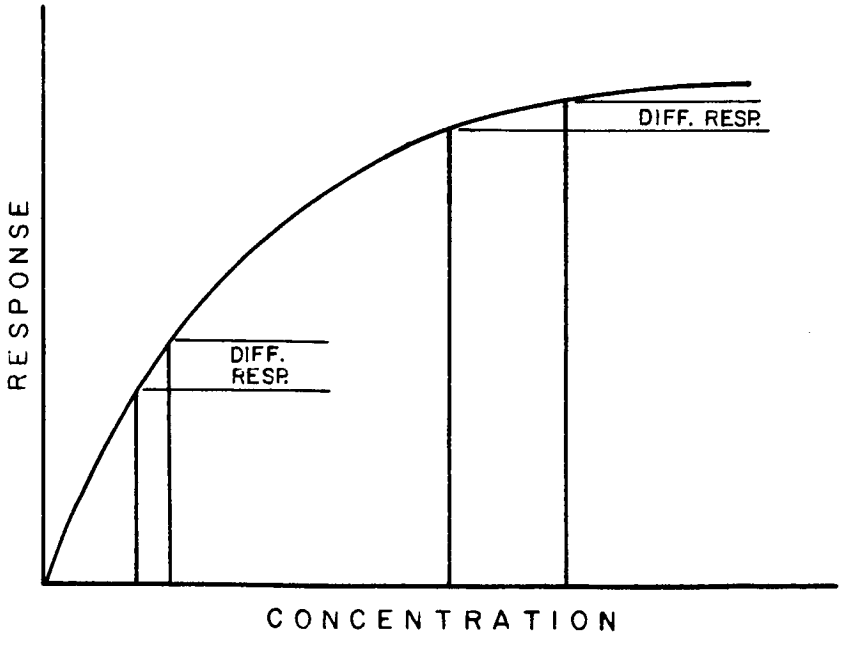

FIGURE 5

DIFFERENTIAL RESPONSE OF NONLINEAR DEVICE

showing a saturation effect, tracer concentration above saturation may cause a decrease in signal, as illustrated in Fig. 5.

When a saturating sensor is used the tracer gas should not be allowed to have a concentration near saturation level, since this could drive both probes into saturation and result in zero signal regardless of the gradient. For such sensors there will be an optimum level of concentration somewhat below saturation and depending on the abruptness of the saturation curve, for which the response to space concentration gradient, dC/dl, will be a maximum. This will give a maximum output for a given probe spacing.

It is interesting to note in regard to these theoretical concentration gradient surfaces that the slowest diffusing tracer gas is the best for gradient sensors since its space gradient is the highest, thus giving larger differences in probe signals. Thus the slow diffusion of freon is as much an advantage for a halogen gradient detector as it is a disadvantage for the simple halogen absolute concentration detector, which is misled by the spurious signals of stagnant halogen pools.

CHAPTER III

STATE-OF-THE-ART DEVICES

We here confine our discussion mainly to state-of-the-art devices for the detection and location of leaks outward from pressurized gas or vapor containing systems. Unless otherwise stated it may be assumed that reference is made to leaks from containers pressurized with air or with air to which has been added an identifying gas or vapor.

This study was conducted by means of visits to sites where leak detection and location of this type is being practiced, letters of inquiry to firms whose operations require such testing, letters of inquiry to manufacturers of instruments for which leak detection capabilities are claimed, by testing purchased or borrowed instruments, and by reading in the current literature.

Methods which seem to be of sufficient currency and effectiveness to be designated state-of-the-art seem to fall into the following five groups:

1. Measurements of rate of pressure change or gas flow.
2. Collection of leaking gas as it leaves the system.
3. Tracer methods.
4. Acoustical methods.
5. Miscellaneous chemical and electrical methods.

Group I

The first of these is perhaps the most simple and, in cases where it is applicable, the best quantitative method. It is, however, a method of detection and measurement but not of location and must therefore be used in conjunction with other methods to find where the leak is. The leak rate in SCIM is given by:

$L = (\partial P/\partial t)528V/T$ where ∂P is the pressure change in atmospheres

∂t is the time interval in minutes

V is the volume of the system in cubic inches

T is the temperature of the system in degree Rankine

This is a simplified standard formula based on the assumption of a constant uniform system temperature. For other conditions, more or less valid formulas are available depending on the extent of the temperature information available. The point of interest to be noted here is that the sensitivity of this method is inversely proportional to the volume of the system. This imposes a severe limitation on its usefulness for large systems or subsystems. An advantage of this technique is the possibility of extending the sensitiveness and accuracy by increasing the time of observation. In other words, the pressure drop method can be made to integrate.

Methods of pressure measurement are too well known to need description here. It is sufficient to remark that many accurate and

sensitive methods are available which lend themselves well to remote indication and automation.

Gas flow meters are also used in certain situations for measuring relatively large leaks quantitatively by metering the volume of gas necessary to maintain constant pressure in the leaking system.

Group 2

The second classification includes the soap bubble method and the immersion method. These are reliable and sensitive methods, capable of semiquantitative interpretation, whose main disadvantage is the labor involved. The former is applicable to missile systems while the latter works well with automobile tire innertubes and other small immersible subsystems. The soap bubble test was perhaps the most universally popular technique found in the search of practical situations.

Group 3

Tracer methods, classification three, includes six main subclasses. These are:

a. Halogen detector methods.

b. Mass spectrometer methods.

c. Thermal conductivity methods.

d. Infrared detectors.

e. Radioactivity detectors.

f. Olfactory detection.

Halogen Detector Methods

The type H-2 halogen detector supplied by the General Electric Company, has been tested and found to be a very satisfactory instrument in the laboratory. A quantitative report of tests indicating its high sensitivity and a critique of the instrument together with suggestions for modifications to make it less vulnerable to background difficulties will be found in Chapter IV of this report.

An important drawback of this instrument is that the tracers to which it reacts are high molecular weight compounds (e.g., Freon 12, Cl_2F_2C, which has a molecular weight roughly four times that of air) which gives rise to difficulties both in mixing these tracers with air (or other pressurizing gas) and in eliminating background. Techniques for mixing are available which can be applied to most situations and various local or general flushing operations can ameliorate the background troubles. At best, however, the halogen detector probably never realizes its full potential sensitivity under use in shop or field environments. In spite of these manifest disadvantages, it should be a very useful instrument if properly employed.

Mass Spectrometer Methods

The mass spectrometer has been developed to a very high state of perfection and has capabilities far beyond mere leak detection. In its present form it can claim sensitivities of the order of 10,000 times

that of its nearest rival. It can respond to any tracer gas. Its main disadvantages are that it must operate with its active parts evacuated which limits the rate at which samples can be ingested with reasonable sized pumping systems. It is definitely practicable in many applications but is not as yet a portable instrument for use on the test floor. Its extreme sensitivity and amenability to tracer selection and variation suggest that it may become so usable in the near future by sacrificing some sensitivity and accuracy for more portability and quicker response times.

The problem of miniaturization and progress which has been made in this direction is covered in Chapter VI of this report.

Thermal Conductivity Methods

Thermal conductivity devices are represented in this study by the thermistor bridges. These are made to respond to the difference in thermal properties of tracer laden gases either by passing a sample successively over opposite members of a balanced bridge or by passing the sample over half the bridge while passing a tare gas (air without tracer) over the other half. While lacking the ultimate sensitivity of the mass spectrometer, these bridges have some of its freedom of selection of tracer and are claimed to be almost as sensitive to helium or hydrogen as the halogen detector is to Freon, in practical situations. Two such bridges, the G.A.S. and the N.R.L. have been tested. They

compare in portability with the halogen detector quite favorably. Test results appear in Chapter V of this report.

Infrared Detectors

The presence of nitrous oxide in a sample of air can be detected by its absorption of infrared radiation. A device of this class is manufactured by the Gelman Instrument Company of Chelsea, Michigan.

Infrared detectors, while operating satisfactorily in certain applications, are not considered to offer any advantages in the present application and therefore were not tested in the course of this study.

Radioactivity Detectors

Radioactive tracer methods are being used with very good success in situations where the advantages seem to outweigh the disadvantages of having to cope with the obvious health problem. These devices are considered beyond the scope of this project and so are only mentioned for the sake of completeness in this report.

Olfactory Detection

A tracer method used successfully for some years by gas manufacturing and marketing concerns and by chemical companies employs the human nose as an extremely sensitive detector for certain sometimes obnoxious sulfur compounds of the mercaptan family. Since methyl mercaptan is detectable when present in air in concentrations

below one part per billion by volume, this method can be used for locating quite minute leaks. The chemistry and physics of mercaptan leak detection is discussed in Chapter IX of this report.

Group 4

Classification four, acoustical methods, includes, first of all, the human ear. Both in its primitive state and when aided by electronic amplifying and background suppression equipment, it is a very useful detector for larger leaks. It is somewhat lacking sensitivity and noise immunity, but leaves very little to be desired in the way of portability.

A recent acoustical device finding some application in the detection and location of leaks is the Ultrasonic Translator an example of which is that manufactured by Delcon, Inc. This instrument responds to a band of acoustical vibrations in the neighborhood of 40 KC by means of a very directional transducer. The electrical output of this transducer is amplified and heterodyned into the audible range by means of a portable transistorized circuit. A leak producing noise in this frequency range within the reception angle of the transducer is announced by a buzz on the built-in speaker, headphones plugged into a jack provided, or may be recorded by connecting a recorder to the output. In its present form the device is relatively low in sensitivity.

Group 5

Miscellaneous chemical and physical methods, classification

five, includes various chemical tapes, smoke producing vapors, catalytic devices for detecting certain reactive gases, and paramagnetic detection of oxygen.

Chemical tapes do not seem to have been put to any use in the field of leak testing of interest herein.

Smoke producing agents, or pairs of agent, are exemplified by ammonia gas and hydrogen chloride (hydrochloric acid fumes) or ammonia and sulfur dioxide. Ammonia would be used as the tracer since it is generally less corrosive than either of the other reagents. This method, while of some utility in a chemical laboratory, seems to offer very little for missile testing.

Catalytic devices have the disadvantage of requiring fairly high concentrations of reactive gases and are subject to the vagaries of catalytic poisoning which can render them insensitive without warning.

The use of the paramagnetic property of oxygen in detecting and measuring its concentration or differences in its concentrations has been highly developed. Presently available units do not seem to be adaptable to portable leak detectors.

In this state-of-the-art survey emphasis has been placed on those devices and procedures which are useful in detecting or pinpointing leaks from systems containing gas under pressure. Several specialized systems which are useful for detecting liquid leaks or leaks

of special chemicals have not been mentioned since they do not seem germane to the problem at hand.

On the following pages, those devices and techniques which seem most applicable to missile leak detection and location are discussed in considerable detail together with results of tests performed in the laboratory.

TABLE II

COMPARISON OF SOME STATE-OF-THE ART DEVICES

Instrument	Tracer	Ideal Sensitivity 100% Tracer	Practical Sensitivity	Remarks
H-2 Halogen Gun	Freon 12	10^{-5} SCIM	10^{-2} - 10^{-4} SCIM	Depends on ambient conditions.
O.U.Halogen Bridge	Freon 12	Less than 10^{-4} SCIM	10^{-4} SCIM	Gradient detector.
NRL Thermistor Bridge	He	10^{-4} SCIM	10^{-3} SCIM	Time derivative. Requires technique.
G.A.S.Bridge	He	2×10^{-3}SCIM	5×10^{-3} SCIM	Time derivative. Requires technique.
Delcon Ultrasonic Translator	None	1 SCIM	1 SCIM	Easily operated. Can perhaps be improved.

CHAPTER IV

HALOGEN GUN STUDIES

The General Electric Model H-2 Halogen Leak Detector

The operation of the halogen gun is based on the fact that the rate of positive ion emission from a hot platinum anode is greatly increased if the air in contact with it contains some organic halogen compound. If, then, the anode of such a diode is heated and supplied with a positive voltage while its cathode is returned to negative through a resistor the voltage from this resistor caused by the flow of positive ion current in the diode can be used as an indication of the concentration of halogen in the air passing over the hot platinum anode .

In the General Electric Halogen Detector tested in the course of these investigations, this voltage is used to provide a signal to an amplifier which in turn drives an appropriate indicator meter or causes a loudspeaker to give an audible signal. Either the voltage or its time derivative may be used to control the amplifier grid by simply inserting a differentiating network into the circuit. The fact that this time derivative of the concentration may be obtained from a space gradient by simply moving the probe at a uniform rate through space, is the basis for the manufacturer's assertion that the automatic mode of operation eliminates the background. Skillfully employed, and but for the disturbing effects of a moving probe, this would give a gradient

sensitive measure which would obviate most of the background difficulty. Unfortunately the requirement for relatively fast uniform probe motion vitiates the advantages of this method considerably.

A simplified schematic diagram of the circuitry is included as Fig. 6. Not shown on the diagram are the pump motor and its associated electrical circuits which produce and control the flow of air to the aspirator which in turn produces the pressure gradient needed to bring the sample into the hot anode diode. The portion of the circuit to the left of the dashed line is contained in the H-2 head or gun, and connected to the rest of the circuit, housed in the main box, by a cable. This cable is hollow and serves to carry the tube which delivers air from the pump to power the aspirator in the gun. The sample is drawn through the concentric platinum cylinder diode, D, by this aspirator. With switch, Sd, in the Hi position the diode circuit is completed through R_1, R_7, and P_2. In the lower sensitivity positions R_7 is paralleled by either R_8 or R_9 to reduce the portion of the signal applied to the grid of T_1. The voltage at the junction of R_1 and R_7, which rises as the result of ionization of halogen tracer in D, is applied to the grid of the amplifier tube through the voltage divider R_{10}, R_{11}. R_{10} serves also to limit grid current in T_1. The capacitor C_1 is in the circuit only when S3 is set for automatic action. This provides the differentiation required to make the instrument respond

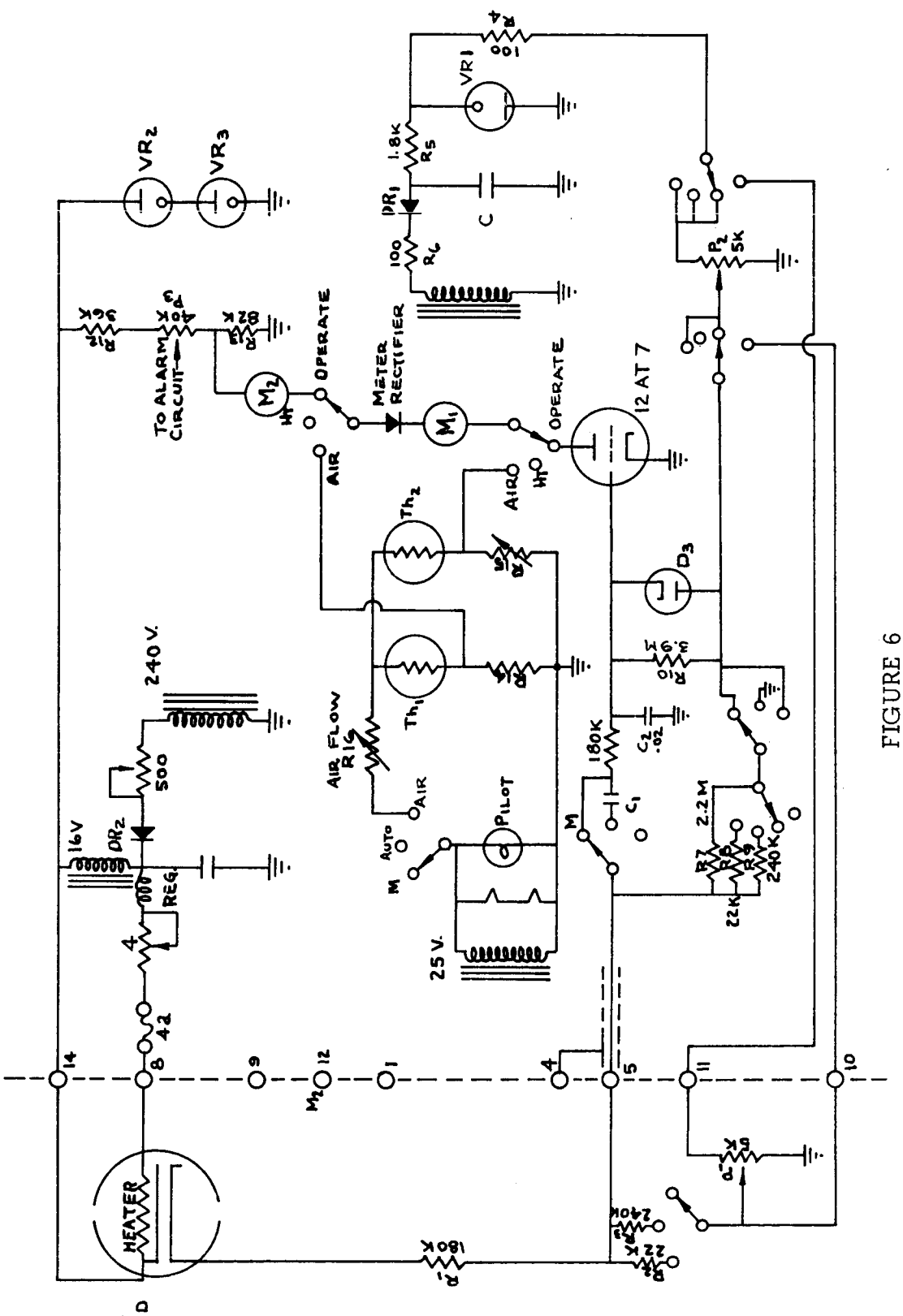

FIGURE 6

HALOGEN LEAK DETECTOR

to time rate of change in halogen concentration (time derivative action). C_2 and R_{10} provide filtering action to prevent tne circuit from being oversensitive to random high frequency noise. D_3 which is half of the 12AT7 connected as a diode provides a rapid discharge path for C_2 when the grid is driven negative. T_1 the other half of the 12AT7 gets its plate voltage from the divider R_{12} R_{13} through the meters M1 and M2 (M2 is really in the head gun) but is shown here for simplicity of drawing. Since the grid voltage of T_1 is determined by the current of the diode (hot anode platinum diode) D, the current measured by the meters is an indication of the halogen concentration (or its time rate of change) in the sample passing through D.

The Air position of S1 and S3 connects M1 to the anemometer bridge, Th_1 Th_2 R_{14} R_{15}, which measures the pump air velocity by its cooling effect on the thermistor, Th_1, which unbalances the bridge.

The heat position of S1 permits use of M1 to set the heater current in the sensor diode, D.

Experimental Evaluation of the Halogen Leak Detector

Various concentrations of Freon 12 in air were prepared in the apparatus sketched in Fig. 7. A measured amount of Freon 12 from graduate 1 was injected into the water filled seven liter container. Air was then admitted, its volume measured by the water displaced into graduate 2. A sample of this known volume ratio was drawn into graduate 3, discharged over the H-2 detector, and the reading noted.

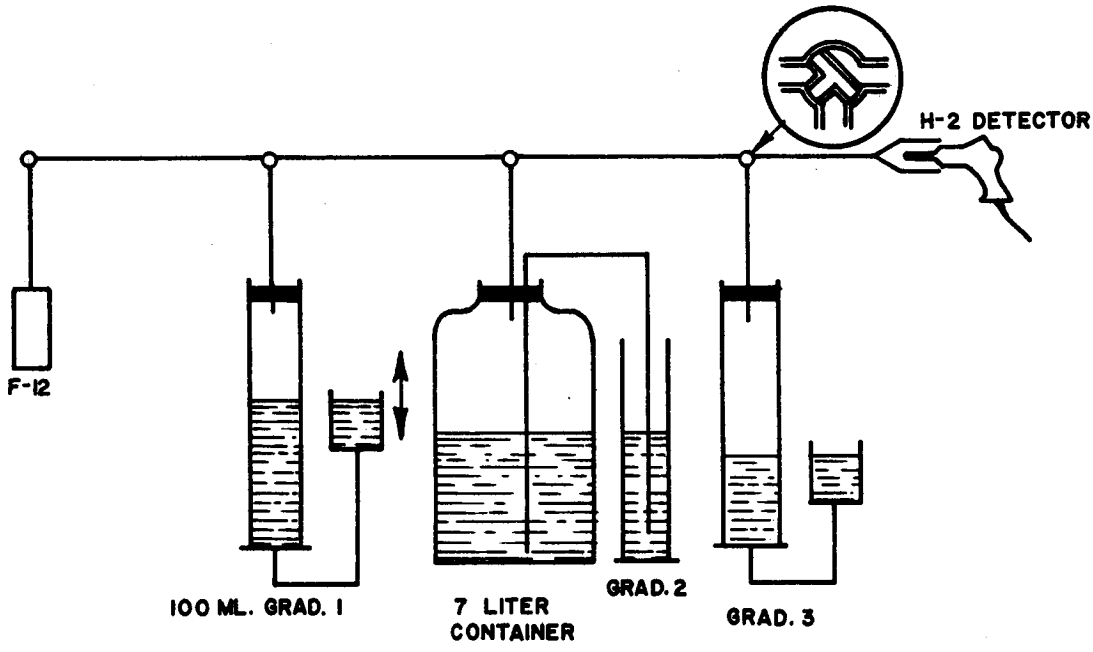

FIGURE 7

APPARATUS FOR DILUTION OF TRACER GAS

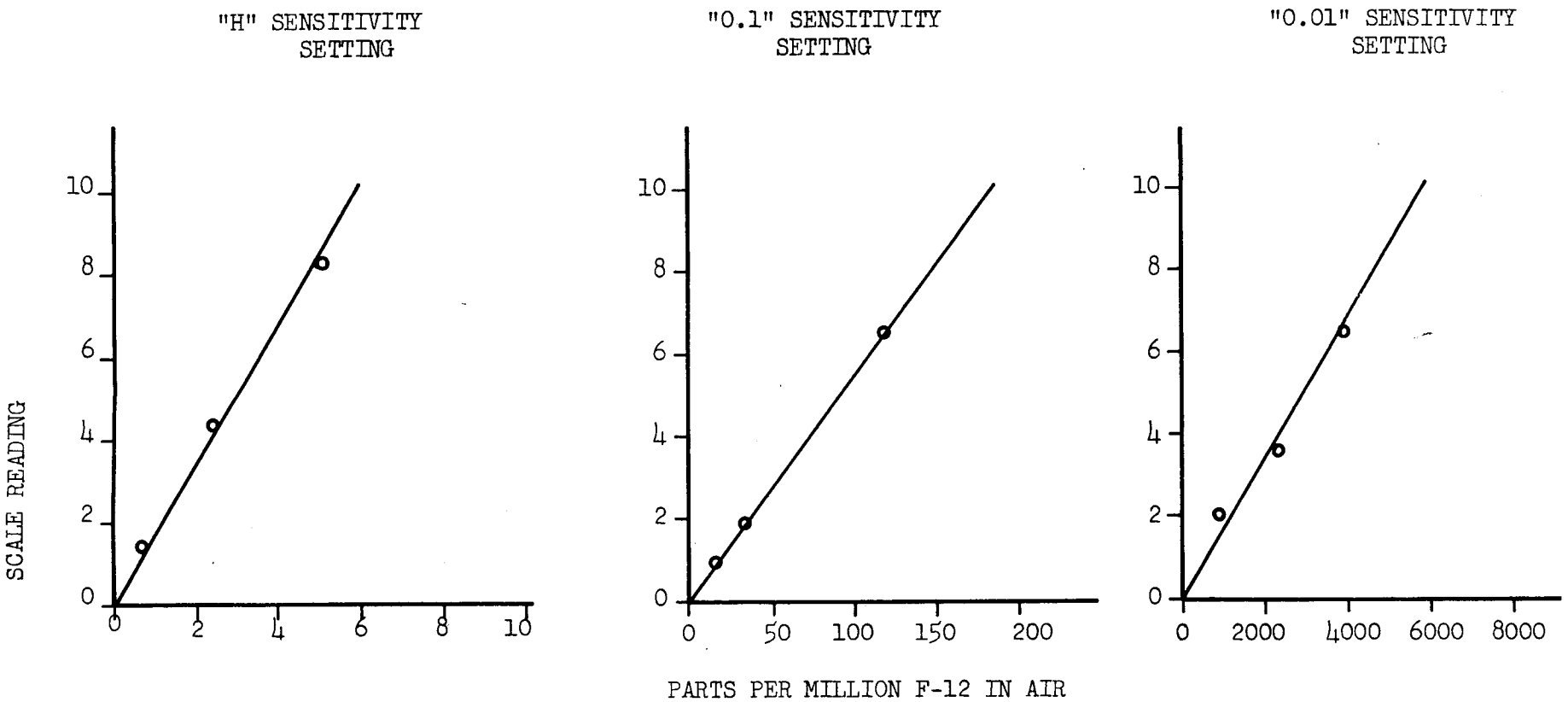

FIGURE 8

SENSITIVITY OF GE TYPE H-2 HALOGEN DETECTOR
SCALE READING VS PARTS PER MILLION F-12 IN AIR

Reduced concentrations were made by drawing smaller amounts from the seven liter sample into graduate 3 and then adding measured quantities of air through the three-way valve.

Tests over the entire range of concentrations were repeated three times, with appropriate care taken in purging the lines with clean air between samplings. The data were reproducible within the range of the dominant error - the reading of the H-2 meter - which varied approximately 5% in repeated readings on the same sample.

It was found necessary to allow 1.5 hours for the H-2 detector to warm up to full sensitivity.

The results of these sensitivity tests are summarized in Fig. 8.

The Halogen Gradient Detector

A halogen bridge gradient detector using standard General Electric hot anode diode sensor elements was put together as shown in Fig. 9. Tests of the basic detector show a linear response for these elements over a range of halogen concentrations from 1 to above 5,000 parts per million of Freon-12. Such a gradient detector should have excellent leak location capability even in the face of very high ambient concentrations.

Fig. 10 presents the schematic of this arrangement. Provision is made to control the anode temperature of the two diodes separately to allow for differences in diode sensitivities. The rest of the circuit

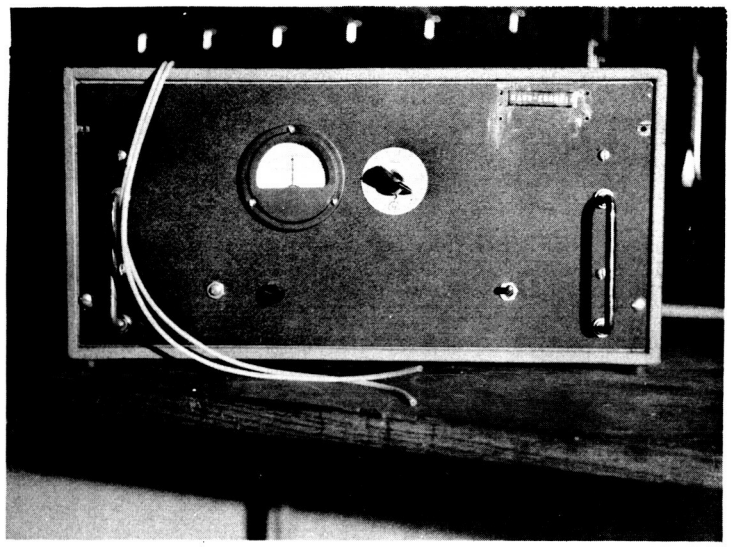

FIGURE 9

OHIO UNIVERSITY HALOGEN
BRIDGE LEAK DETECTOR

is a high gain differential amplifier and chopper arrangement to obviate the effects of drift, and a regulated power supply for added stability.

D_1 and D_2 are the hot anode diode detecting units. When a halogen gas is in contact with the anode of either diode, positive ions are emitted. Ionization in D_1 gives rise to a current through the divider formed by R_1 and R_3. Ionization in D_2 causes current to flow through R_2 and R_3. Equal ionization in both diodes causes the bridge formed by D_1, D_2, R_1, R_2, R_3 to be balanced. Under this condition there will be no potential difference between Tp_1 and Tp_2. Whenever, on the other hand, there is a difference in the amount of freon (halogen) in the diodes there will be an unbalanced bridge and a potential difference will appear between Tp_1 and Tp_2. This potential difference is applied to the two grids of V_1. These grids are also fed with two 60 cycle voltages which are 180° out of phase with each other. For simplicity one grid will be referred to as the 180° grid and the other as the 0° grid. These two signals are of equal amplitude when the bridge is balanced and no output results on the cathode of V_1 due to the cancellation of the currents combined in the resistor R_4. Whenever an unbalance occurs due to a higher current in D_1 than in D_2, a 180° voltage will appear on the cathode of V_1. If D_2 has the higher current (higher concentration of halogen) the resultant voltage will be 0°. The amplitude will be dependent on the amount of unbalance in the bridge in either sense. This voltage is taken as the cathode of V_1

across R_4 and coupled through C_3 to the grid A of V_2. The resistor R_7 is the grid return for section A of V_2. The unbypassed cathode resistor R_8 provides feedback to reduce the gain of the amplifier circuit of V_2 section A. The signal is coupled from the plate of section A of V_2 through capacitor C_4 to the grid of section B of V_2. This stage is biased by the combination of R_9 and C_5 in its cathode. C_5 reduces degeneration in this stage. The output of this stage is taken from the plate by the divider consisting of capacitors C_6 and C_7. At the junction of these capacitors a synchronous detector is connected. The output of this detector is across R_{13} the wiper of which is connected to a 150-0-150 microampere meter movement. This is the visual readout of the halogen bridge detector.

Section B of tube V_3 is a paraphase amplifier which amplifies a portion of the 60 cycle filament voltage taken from the junction of the resistors R_{21} and R_{20}. The outputs of this amplifier are taken from its plate and cathode. The two outputs are 180° out of phase with each other. The variable resistor, R_{17}, the phasing control, allows any intermediate value of phase between 0° and 180° to be taken from section B of V_3 and applied to the grid of section A of this tube. V_3 serves as a phase splitter or phase inverter for deriving the two 60 cycle voltages for the differential cathode follower, V_1. With the phase control properly set the contact of the chopper will close when the

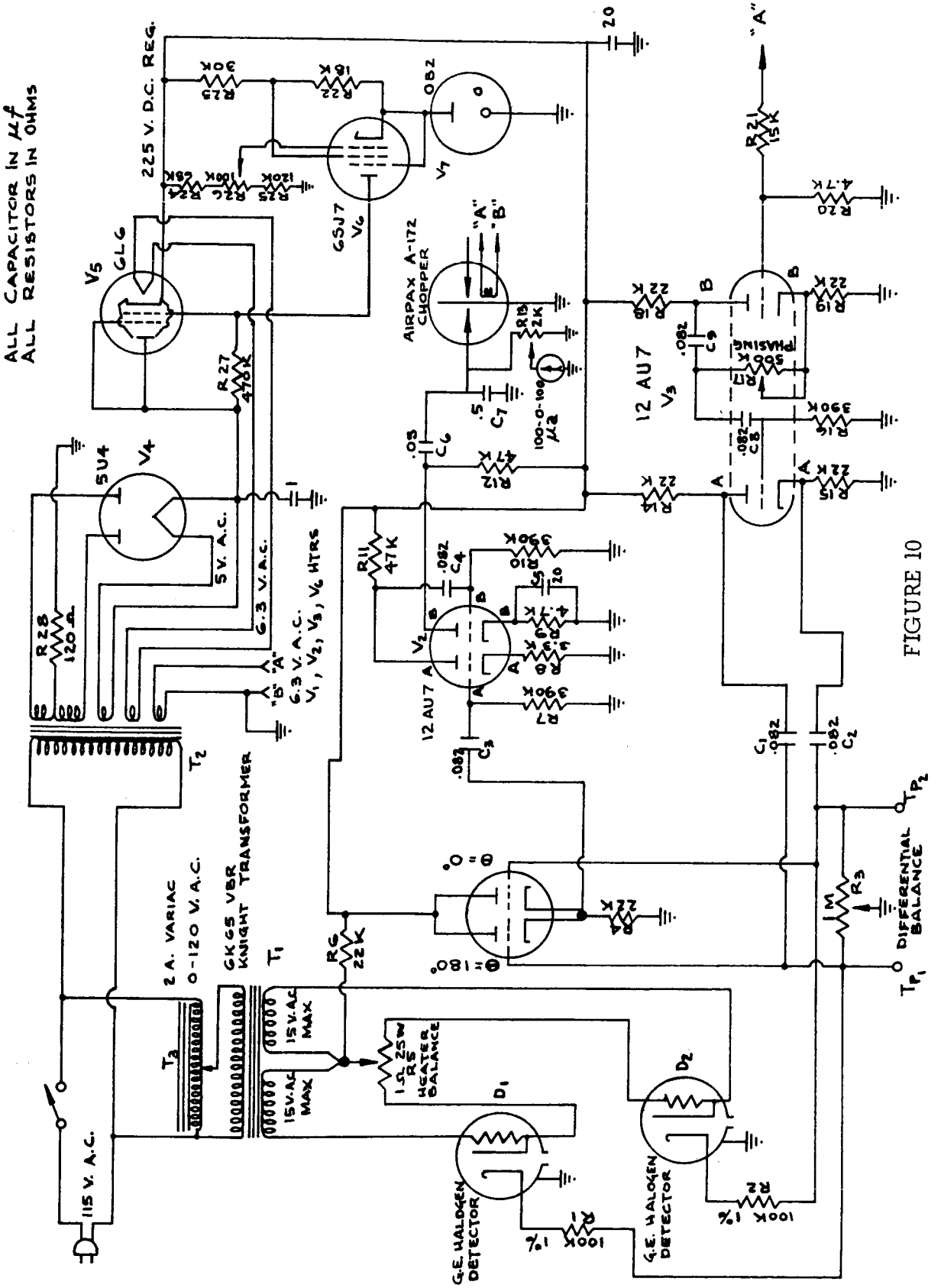

FIGURE 10

OHIO UNIVERSITY HALOGEN BRIDGE
LEAK DETECTOR SCHEMATIC

60 cycle voltage applied to its contacts passes through zero and open on the next half cycle when a zero voltage crossing occurs. The result will be half wave rectification of the output signal due to one half cycle being shorted to ground. The output polarity will depend on which signal is present in the output, 0^{o} or 180^{o}.

The heater supply for the hot anode diodes is controlled with a 2 ampere Variac feeding a dual 15 volt secondary transformer T_1. A balance potentioneter, R_5, is used to balance the heaters of the two detectors for more matched operation.

The power supply for the detector is an electronically regulated unit of straightforward design.

Preliminary tests show that this detector is capable of locating a leak of less than $6x10^{-5}$ SCIM in an ambient background contamination capable of causing half scale deflection on the intermediate range scale of the standard H-2 General Electric halogen detector. In the same circumstances, the standard halogen detector showed no signal variation near the leak and was completely incapable of detecting it.

Correlation of Tests on General Electric H-2 Halogen Detector Naval Research Laboratory Thermistor Bridge, Model 410, and Ohio University Halogen Bridge

A series of tests have been conducted to obtain comparable evaluations of the Halogen detector, the thermistor bridge, and the

halogen bridge (gradient detector). The absolute halogen detector was tested both in the state furnished by the manufacturer and with the addition of a flushing hood. The unhooded instrument (H-2) served as a standard for the measurement of background contamination levels. A variable standard leak (General Electric Model LS-20) served as a source of halogen gas (Freon-12). The test setup is shown in Fig. 11.

The following procedure was employed to activate the test conditions. A small amount of freon was allowed to escape into the air stream of the fan. The amount of freon contamination was then checked inside the duct with the standard General Electric detector (H-2) and the flow of freon adjusted until the contamination reached the desired level for the test.

The first test was run with a contamination level which registered about half scale on the H-2 detector with the sensitivity switch set at 0.1. The standard leak was set at 0.5 oz. per year (3.5×10^{-4} SCIM). The standard halogen (absolute concentration level) detector gave no indication of the leak at all. Some slight indication was available when the probe was placed directly on the leak, but this could not be done without knowledge on the part of the operator as to the position of the leak. Contamination variation away from the leak completely masked the concentration variation at the leak. The hooded detector (described in Chapter IX) dropped the background reading by a factor of about 2 by

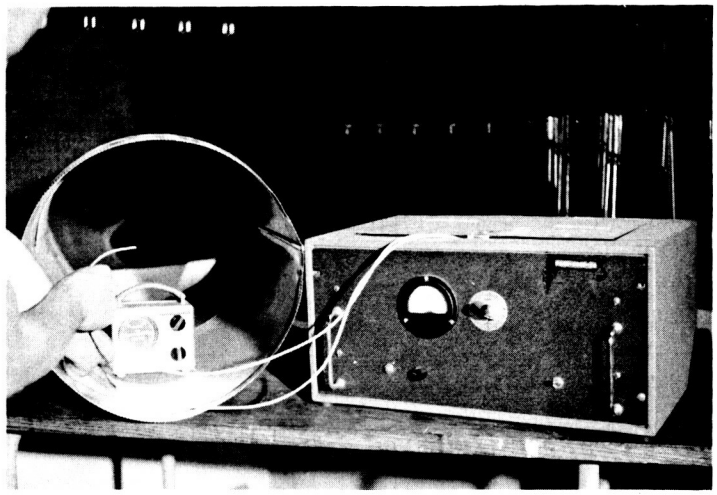

FIGURE 11

TEST SET-UP FOR HALOGEN
BRIDGE STUDY

means of a gentle air flush from a nitrogen tank. Some slight deviation of the reading was noted during exploration but this was not enough to indicate a leak to an operator ignorant of its position. Even with the shroud contamination gradients still masked the leak pattern. While some full-scale readings were obtained at the leak, these were non-repeatable and erratic readings were obtained elsewhere.

The Naval Research Laboratory Model 410 thermistor bridge operated in its differential mode was found sensitive to the background contamination when one probe was outside the duct and the other inside. The leak itself was, however, undetected by this instrument. Additional tests showed that a leak of 3 oz. per year (21×10^{-4} SCIM) gives a very weak response even without the contamination problem. A reading of 4 (full scale 20) was obtained in this case. It must be pointed out that this instrument is much more sensitive to hydrogen and helium than it is to halogen.

Tests were conducted on the halogen gradient bridge with the sensitivity set at 3/4 of full and with the heater control at 116. With both tips together in the contaminated area the instrument was found to be zero with variations not exceeding 10% of full scale deflection over the range of concentrations presented. If either probe tip was taken out of the contaminated area the instrument immediately indicated full scale deflection in the direction for detection by the probe left in the

contaminated area. Stabilization of the unit was found to be slightly touchy and the sensitivity control was backed off to 1/2. This improved the stability considerably.

Two test procedures were used with the leak set at 0.5 oz. per year (3.5×10^{-4} SCIM). When the two tips were held together and passed across the leak area the needle of the instrument was found to deflect first in one direction and then in the other depending on which tip passed the leak first. The deflection was about 50 to 60 (full scale 100) which seems quite adequate for detection and location of the leak. The second method consisted of placing one probe tip about 1/4 inch ahead of the other as shown in Fig. 12. This was found to give excellent results. Approaching the area of the leak resulted in full scale deflections of the instrument each time. It was possible to pinpoint the leak location simply by exploring with the probe with the operator's eyes on the instrument. Reversing the relative positions of the probe tips reversed the direction of the meter deflection.

The contamination level was changed to about full scale on the H-2 detector on the 0.1 setting of the sensitivity control. It was found that the bridge had to be readjusted for this new contamination background. This was ascribed to accidental differences in the two hot anode detectors, which are not, of course, designed or selected to be used in pairs and therefore should not be expected to match. This

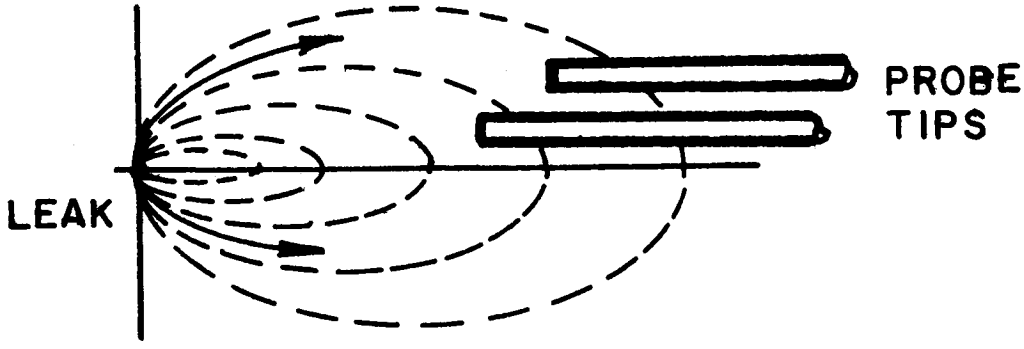

FIGURE 12

ARRANGEMENT OF PROBE
TIPS ON HALOGEN BRIDGE

problem is trivial and might be solved either by obtaining matched diodes or by providing electronic feedback compensation circuitry. The zero shift was small enough so that the leak could be detected even before the instrument was rezeroed. It was found that the bridge responded very well in the higher background contamination and the leak of 0.5 oz. per year (3.5×10^{-4} SCIM) was still easily detectable with full scale readings near the point of leak.

The background was returned to its original value (about half scale on the H-2 with sensitivity setting 0.1) and the leak rate was decreased from 0.5 oz. per year (3.5×10^{-5}). The results are given in Table III.

TABLE III

Leak Rate SCIM x 10^4	Meter deflection probes together	Meter deflection probes echelon	Remarks
3.5	full scale sweep	full scale sweep	
2.8	full scale sweep	full scale sweep	
2.1	80% full scale	full scale sweep	
1.4	60% full scale	80% full scale	
0.7	30% full scale	40% full scale	Lower limit of Method 1

Operation of Halogen Detectors

For purposes of reference and comparison, simplified instructions for operation and use of the General Electric Halogen Detector and of the Ohio University Halogen Gradient Detector have been placed in Appendix A.

CHAPTER V

THERMISTOR BRIDGE

The thermal conductivity sensor depends for its action on the fact that different gases or different concentrations of a gas in air have different cooling effects on a heated electrical resistance element over which they pass. This cooling effect establishes the temperature of the element for a given electrical power input and gas rate of flow. The temperature, in turn, establishes the resistance of the element.

If four such sensing elements are connected as a Wheatstone bridge, a very sensitive device for the detection of either concentrations or concentration gradients results. If absolute concentrations of helium or hydrogen are to be measured, two elements of the bridge will have the tracer laden air drawn over them while the other two elements are furnished with ordinary air. The higher conductivity of the helium or hydrogen laden air will cool two of the resistors and thus unbalance the bridge. The potential resulting can be amplified and used as an indication of the amount of tracer present.

If, on the other hand, samples of gas from two nearby points are drawn over opposite pairs of bridge elements, the unbalance will result from the difference in concentrations at the nearby points. Thus the unbalance will be proportional to the concentration space gradient.

In a third mode of operation, the sample may be drawn successively

over the four bridge elements. This will cause temporary unbalance of the bridge whenever the concentration at the point of investigation is changing with time. If the probe is being moved continuously, space gradients are converted into time gradients. This necessity for continuously moving the probe in order to read space gradients may bring about a need for more skill on the part of the operator who is likely to respond instinctively to a high gradient indication by abruptly stopping the motion and thus having the indication disappear. Training, however, should make this mode useable by most operators.

Several materials are used as active elements in these bridges. Tungsten and platinum have long been favorite materials. These have resistivities which increase with temperature. Currently, semiconductor materials which decrease in resistivity as temperature increases are being used. The magnitude of the change is much greater with the semiconductors than with the metals. These semiconductor temperature sensing elements are called thermistors and find many applications based on the very high temperature dependence of their resistances.

The Naval Research Laboratory Thermistor bridge detector obtained in connection with this project for testing is described and its sensitivity to various tracer gases is reported in NRL Reports 5647 and 5807. Fig. 13 summarizes the sensitivity figures given in these reports. Tests carried out in the Ohio University laboratory

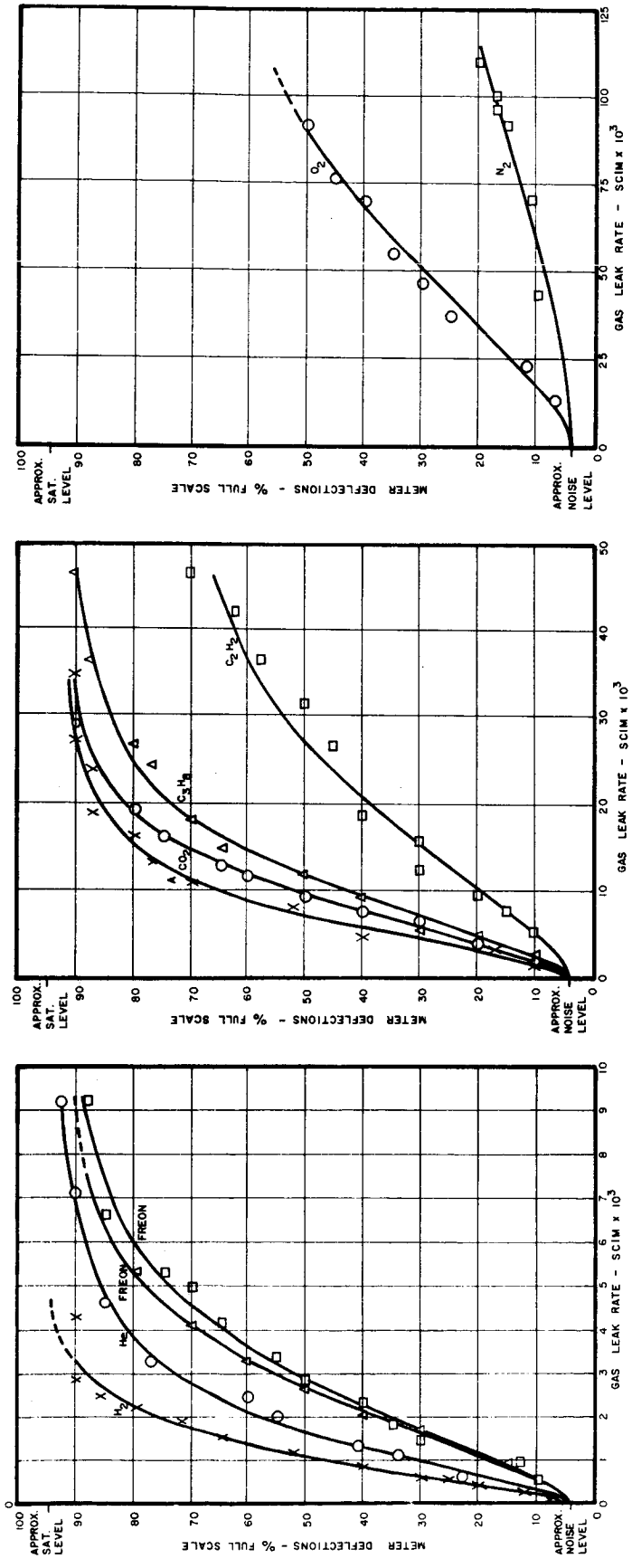

FIGURE 13

SENSITIVITY FIGURES FOR
NRL THERMISTOR BRIDGE

confirmed the data of Fig. 13.

It is to be noted that the thermistor bridge shows saturation effects. This limits the tracer concentration which can be used and hence the sensitivity, as pointed out in the discussion of gradient detector problems. A possible solution to this difficulty lies in a circuit redesign which would provide constant thermistor temperature through a feedback system. The output would then come from measurement of the required feedback effort. Such a system would give linear response over the entire range of tracer concentration. Fig. 14 shows the present NRL bridge circuit.

Functional Description

The sequence of events which occurs when a gas leak is detected can be followed by referring to Fig. 14. The gas passing over thermistors T_{s1} and T_{s2} changes the bridge balance if the thermal conductivity is different from air. The unbalance produces an output voltage from the bridge which is applied to the input filter. The series condenser, C, prevents any constant or slowly changing dc bridge voltage from reaching the chopper, thus eliminating the need for manual balancing of the bridge.

The two shunt condensers, C_3 and C_4, provide a long decay time constant to filter out random noise of frequency higher than a few cycle per second, and also to provide a long holding time to permit

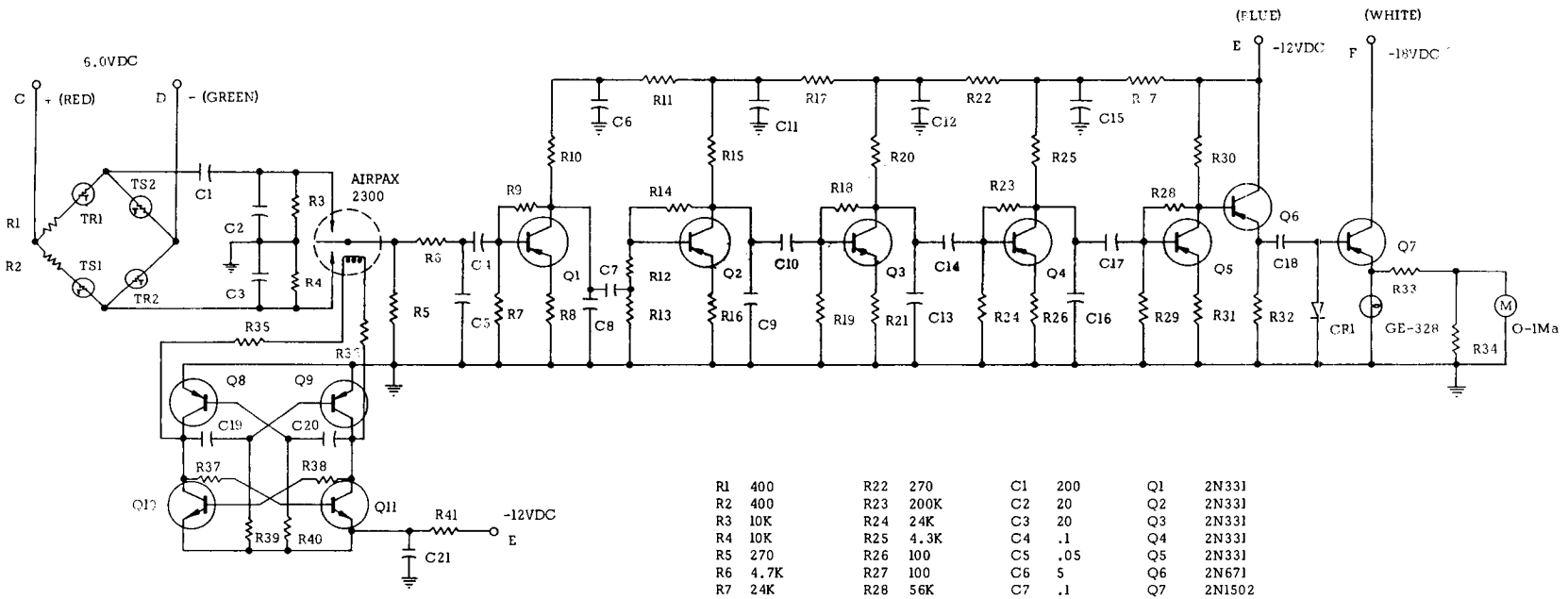

FIGURE 14

SCHEMATIC OF NRL BRIDGE

R1	400	R22	270	C1	200	Q1	2N331
R2	400	R23	200K	C2	20	Q2	2N331
R3	10K	R24	24K	C3	20	Q3	2N331
R4	10K	R25	4.3K	C4	.1	Q4	2N331
R5	270	R26	100	C5	.05	Q5	2N331
R6	4.7K	R27	100	C6	5	Q6	2N671
R7	24K	R28	56K	C7	.1	Q7	2N1502
R8	100	R29	27K	C8	.05	Q8	2N425
R9	220K	R30	1K	C9	.05	Q9	2N425
R10	5.6K	R31	100	C10	.1	Q10	2N388
R11	4.3K	R32	240	C11	10	Q11	2N388
R12	2.7K	R33	1.8K	C12	10		
R13	22K	R34	1.8K	C13	.05	CR1	1N54A
R14	220K	R35	100	C14	.1		
R15	5.6K	R36	100	C15	100		
R16	100	R37	10K	C16	.05		
R17	2.2K	R38	10K	C17	.1		
R18	220K	R39	10K	C18	8		
R19	24K	R40	10K	C19	.15		
R20	5.6K	R41	150	C20	.15		
R21	100			C21	100		

observation of the indication resulting from the relatively short pulse type signal. These condensers are of the nonpolarized types.

The signal is modulated at a 400-cps rate by the chopper, Airpax 2300. The resulting ac signal is applied to the first transistor amplifier, Q_1, through R_6 and C_4. All transistor amplifier stages Q_1 through Q_5 are very similar and have bandpass characteristics centered at 400-cps. The output of transistor Q_5 is directly coupled to the base of transistor Q_6 which is an emitter follower.

The output of the emitter follower is demodulated by a germanium diode, CR_1, and applied to the base of Q_7 as a dc potential. The output of Q_7 is used to drive a GE 328 lamp and is passed through multiplier resistor, R_{33}, to the indicator meter which is shunted by resistor R_{34}.

The modulator, an Airpax 2300 chopper, is a low noise 400-cps unit which is driven by a 400-cps multivibrator with a balanced output. The multivibrator consists of Q_{10} and Q_{11} which are pnp transistors. Q_8 and Q_9 are flip-flop connected and also serve as collector loads for the multivibrator. Q_8 and Q_9 are npn type transistors from which the drive power is obtained for the chopper.

Advantages

Some advantages of the thermistor bridge thermal conductivity detector include:

1. The use of low molecular weight gas such as hydrogen or helium increases the diffusion rate in air by a factor of about 7, reducing the need for flushing blind ducts and helping considerably in solving the contamination and pooling problem. These gases also increase the leak rate for a given leak and thereby make it easier to locate.
2. The built-in bridge circuit configuration is adapted for gradient sensing.

Some disadvantages of the thermistor bridge include:

1. In its present form it is not as sensitive as the halogen detector and therefore can not be used to locate very small leaks.
2. In its present form it saturates which limits its sensitivity as a gradient instrument.

Operation of Thermal Conductivity Bridges

For purposes of reference and comparison, simplified instructions for operation and use of the NRL Thermistor Bridge and the G.A.S. Thermistor Bridge are included in Appendix A.

CHAPTER VI

MINIATURIZED MASS SPECTROMETER

Introduction

This chapter presents a discussion of this approach along with some of the more pertinent characteristics and performance data of similar equipment which already exists. The availability of this similar hardware provides a firm foundation on which to base the anticipated performance characteristics for the final miniaturized equipment.

A mass spectrometer is a very versatile tool for the analysis of gases, liquids, solids, and plasma. When properly designed it can handle wide ranges of concentration accurately and with very fast response. Of initial interest is the sensitivity of a mass spectrometer leak detector. It is easily possible to detect a leak of the order of 10^{-10} standard cc per second (4×10^{-10} SCIM) (4×10^{-13} SCIM with refinements) which is 10^4 times more sensitive than the commercially available portable halogen leak detector. Also of prime importance is the fact that if helium or some other light tracer atom is used in conjunction with the mass spectrometer leak detector it will diffuse away from the leak at a much greater rate than the relatively heavy halogenated compound tracer when there are none actually present. Detection can be made from any direction and helium being much lighter

than air will remove itself from the vicinity of the leak without the necessity of a flushing operation.

The research activity has been associated with determining if this type of instrument is feasible for applications where size, weight, reliability, and fast response are of extreme importance.

Mass Spectrometers

The components of a mass spectrometer which are of principal concern are the ionization and acceleration region, analyzer section, ion detector with associated electronics, and vacuum pumping apparatus.

A typical commercial mass spectrometer leak detector is the General Electric Type M-60. Helium is used as a tracer gas because it is light and hence diffuses rapidly into a leak, it is relatively inexpensive and available, and it cannot explode or catch fire.

The helium is applied to the interior of the system to be treated at a higher pressure than the pressure surrounding the system. The air sample is then taken from the outside surface of the equipment through a "sniffer" and introduced into the spectrometer. Another procedure is to evacuate the system to be tested and spray a jet of helium over the suspected area. If a leak is present some of the helium passes through to the interior and is then pumped into the spectrometer. It is best to pressurize pressure systems and evacuate vacuum systems when testing.

The leak detector operates on the mass spectrometer principle. The mass spectrometer has a special electron tube with electric and magnetic fields that sort out ionized gas molecules according to their mass to charge ratio. The selective properties of the mass spectrometer make it ideally suited for leak detection. For helium detection the spectrometer tube should give nearly a zero output for air and residual gases and respond only to the helium tracer gas. This sorting of helium from the other substances depends upon the forces acting on charged particles traveling in a magnetic field.

A magnetic field exerts a force on a moving ion. After an ion enters a magnetic field perpendicularly it is forced to travel a curved path approximating the arc of a circle within the field. When it emerges from the magnetic field it is thus traveling at an angle with respect to its original direction. This angle, and the radius R of the arc of the circle within the field depend upon the strength of the magnetic field, the accelerating potential, the mass of the ion, and the charge of the ion in the following manner:

$$M = \frac{m}{q} = K \frac{R^2 B^2}{V}$$

where q = charge of the ion in electron charge units

m = mass of the ion, in gram atomic weight units

M = "specific mass," in gram atomic units per unit charge

B = magnetic field strength in gausses

V = accelerating potential in volts

R = radius of curvature of the ion path, in centimeters

K = constant of proportionality, equal to 4.8×10^{-5}

In the spectrometer tube of the leak detector the magnetic field B is fixed, and the accelerating potential V is adjusted so that helium ions travel with a desired radius R. All other ions have a different mass to charge ratio;therefore the radii of their paths are different. A collector is placed so that only those ions traveling along one curve, that of the helium ions, will enter it.

Gas molecules of the air sample from the object under test enter the spectrometer tube and flow into the ionization chamber. Electrons emitted from the hot tungsten filament are beamed into the chamber, where they bombard the gas molecules and produce positive ions by collision. The electron beam is formed by the difference in potential between the filament and ionization chamber.

An ion repeller plate inside the ionization chamber is positive by a few volts with respect to the chamber. This plate repels the positive ions formed by bombardment, and gives them an initial acceleration. The main accelerating field is between the ionization chamber and exit plate. The exit plate is at ground potential; it is thus negative with respect to the positive ions, and it attracts them. Two focus plates

between the ionization chamber and exit plate serve to direct the ion beam through the slit in the exit plate. The result is the formation of a narrow beam of ions from the air sample.

The ion beam passes at right angles into the field of the analyzing magnet. After leaving the magnet those ions with a particular mass to charge ratio (that of helium) will follow the correct straight path into the collector. Ions from all other gases have a different mass to charge ratio; therefore their curvature while in the magnetic field will be different and they will not enter the collector.

The selective property of the spectrometer tube is improved by the addition of a suppressor plate. In normal operation ions of a larger mass to charge ratio than helium collide with other ions and lose some of their energy. These heavier ions then move more slowly than normal, and tend to travel curved paths of smaller radius than normal through the analyzing magnet. This causes them to travel toward the collector. If these undesirable ions reach the detector plate, they will produce an output reading called background. This background is objectionable, because it decreases the ultimate sensitivity of the leak detector; since any helium output must be superimposed on the background, the background is minimized by the suppressor plate. It has a positive potential that repels the slowed-up ions of mass higher than helium, deflecting them from the collector. The helium ion current is then

amplified for display.

The simple principles of operation as outlined above made no mention of the focusing properties of the analyzer. This is a subject normally referred to as positive ion optics and is fundamental to the field of mass spectrometry. As the ion beam enters the analyzer section the ions have a certain energy and direction distribution. The purpose of focusing is to assure that the atoms of one particular mass strike the collector regardless of small variations in direction or energy.

This subject is well developed in the literature[1] and only reference will be made to it here. The discovery of the focusing properties of the so called sector fields has led to the widespread use of sector magnets[2] (normally 60^{o} or 90^{o}). This applies to direction focusing in homogeneous magnetic fields. Direction focusing in the radial electrostatic field which exists between the plates of a cylindrical condenser is also discussed in reference 2. Double focusing in consecutive field combination can be accomplished by letting, for example, the image formed by a radial electrostatic field serve as the object for a following magnetic field.[3] Bleakney and Hipple[4] showed that the combination of crossed uniform magnetic and electric fields possessed perfect double focusing in the plane normal

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[1]Superscript numerals refer to references at the end of this chapter.

to the magnetic field Johnson and Nier[5] have described as a double focusing combination possessing, at a given point, both second-order direction focusing and first order velocity focusing. Focusing in non-uniform fields has been discussed by Lee-Whiting and Taylor.[6] In the detection of leaks we are associated with abundance measurements and the elaborate focusing properties associated with high resolution instruments are not so critical.

Positive ion sources take many forms depending upon the element to be studied and the nature of the study. Ideally the source should supply a monoenergetic ion beam which is rich in the desired ions and free of undesired ones. In practice it is frequently necessary to relax one or the other of these requirements in order to improve the performance of the source in a particular desired respect. Only the ionization of gas samples will be considered.

In early work the gas discharge was the principal source of positive ions. After formation, the ions are accelerated toward a perforated cathode, through which they pass, en route to the analyzer, with a wide range of energy and degree of ionization. Obviously the wide energy spread among the ions is a serious disadvantage, since it limits the use of the source to velocity or double focusing analyzers.

Nier[7] is largely responsible for the modern electron impact source. Here the sample material, in form of a gas or vapor, passes

through an opening to the ionization chamber, where it is bombarded by a beam of electrons whose energy may be varied to secure maximum ionization efficiency. For most singly charged ions the ionization cross-section reaches a maximum in the range 70-90 volts.

The positive ions so formed are urged downward by a weak drawing out field between a repeller and the opposite wall of the ionization chamber and emerge from the latter through a slit which is parallel to the direction of the electron beam. They are then accelerated and pass through a slit to the analyzer region.

The ions arising from the electron impact source are nearly homogeneous in energy. This is primarily due to the fact that since the ionizing beam is narrow the ions are created more or less along an equipotential surface and experience similar drawing out accelerations. Usually the width of the electron beam is reduced even further by use of an externally applied magnetic field parallel to the direction of the electron path. Electrons which would otherwise diverge from the specified path are in this way constrained to describe close helices about it. This generally results in an increase in the intensity of the positive ion beam as well as an improvement in the monochromaticity which is sought. A homogeneity in energy of 0.05 eV can be achieved.[8] The electron impact source is the most widely used in mass spectrometry. A new pulsed ion source of this type will be discussed

below in connection with a proposed miniaturized leak detector.

The vacuum spark source[9] of positive ions is incompatible with our requirements due to an extremely large energy spread.

The Philips Ionization Gauge (P.I.G.) type source employs a cold discharge in a magnetic field and possesses an energy spread of approximately 25 eV[10]. It has the advantage of being able to operate in the relatively high pressure range of 10^{-4} - 10^{-3} mm of Hg.

Detection of positive ions in a mass spectrometer is accomplished by standard amplification techniques. Direct current amplifiers,[11] vibrating reed electrometers,[12] electron multipliers[13] and scintillation detectors[14] are used for varying applications. The vibrating reed electrometer is extremely stable relative to the D.C. amplifier and the electron multiplier has extreme sensitivity and fast response as its principal merits. The former permits the detection of single ions and the latter allows a rapid scanning of the mass spectrum. A relatively recent magnetic electron multiplier will be discussed below in connection with a time of flight mass spectrometer. In the scintilation detector the ions are accelerated to a relatively high energy and projected upon a luminescent material. The resulting photons are lead via a light pipe to a conventional multiplier tube and amplifier. This system has a short response time. Disadvantages are that the dark current in photomultiplier tubes is approximately two orders of magnitude

greater than in electron multipliers, the luminescent efficiency of phosphors deteriorates under prolonged ion bombardment, and phosphors display a strong mass discrimination.

The last component to be investigated is the vacuum pumping apparatus. A minimum vacuum of 10^{-5} mm Hg must be available to allow the ions to traverse the path through the analyzer and to facilitate long filament life if an electron impact ion source is employed. This is normally accomplished through ordinary fore-diffusion pump equipment. Since this type of equipment is not easily miniaturized without a large decrease in performance, a Vacion getter-ion type pump will be described below which has more than sufficient capabilities for our application.

The previous discussion of positive ion optics concerned analyzers which would be incorporated into mass spectrometers of the so called "deflection" type. The other important category is the TOF or time-of-flight mass spectrometer. Time-of-flight instruments measure the time required for an ion to traverse a certain specified distance. This may be done by a direct timing mechanism, employing pulsed ion sources and detectors or by subjecting the ions to radio-frequency fields. In either case the apparatus selects from the ion beam, those ions having a certain velocity. If the velocity of the ion be characteristic of its mass, as in the case of singly charged ions

which have fallen through the same potential, such a velocity filter may be used to effect a mass analysis.

In certain other mass spectrometers the ions describe circular paths in homogeneous magnetic fields, with the cyclotron frequency.

$$f = \frac{He}{2\pi Mc}$$

where H = magnetic field strength in oersteds

e = electronic charge in statcoulombs

M = mass in grams

c = velocity of light in cm per second

This is independent of velocity, but linearly dependent upon reciprocal mass. Here the mass is determined by measuring the cyclotron frequency, usually in terms of a high harmonic. Instruments of this type are referred to as cyclotron resonance mass spectrometers.

Advances in the techniques for the generation of very short electrical pulses provided the basis for pulsed beam mass spectrometers developed by Stephens[15] and Cameron and Eggers[16].

These instruments consist essentially of a comparatively long drift tube, with a source of ions at one end and a detector at the other. The source emits short bursts of ions, homogeneous in energy or momentum. These traverse the drift tube to reach the detector, which is sensitized for a brief instant to register their arrival. Since ions of different mass arrive at the detector at different times, the accurate

measure of the time between activating the source and sensitizing the detector gives information concerning the mass of the ions being detected.

If L is the length of the drift tube, the transit time for singly charged ions of constant energy Ve, is

$$t = L(M/2Ve)^{1/2}$$

and for those of constant momentum, P, is

$$t = LM/P$$

If the collector is sensitized for a period Δt at time t, the resolution becomes, for constant energy ions,

$$\frac{\Delta M}{M} = \frac{2\Delta t}{t}$$

and, for constant momentum ions,

$$\frac{\Delta M}{M} = \frac{\Delta t}{t}$$

It will be noted that the arrangement employing a constant momentum source possesses, in theory, a factor of two advantages over the constant energy type. However, it is not Δt which in practice limits the resolution, but rather ion thermal energies. When these are taken into consideration,[17] the advantage may swing in the other direction.

It was pointed out in descriptions of the early models of this type of instrument that they would allow a rapid panoramic display of the

entire spectrum, and this feature was emphasized by the use of cathode-ray oscilloscopes as display units. A refined version of this instrument will be discussed below in connection with our miniaturization study.

Energy gain mass spectrometers are instruments in which certain ions with a particular initial velocity are accelerated by periodically varying fields to the extent that they are able to overcome a d.c. potential barrier and reach a collector. Other ion experience insufficient acceleration in the r.f. field to surmount the final retarding voltage.

Bennett[18] has developed a mass spectrometer of this sort in which the ions created by electron bombardment, fall through a modest fixed voltage before entering a three-stage resonance accelerator. Each stage consists of three grids and to the middle one in each case is applied the r.f. potential. As the acceleration experienced by an ion in each stage depends upon its velocity and the phase at which it enters, the three stages can be so placed relative to each other that ions with one particular initial velocity and phase experience a much greater cumulative acceleration than do any others. These, as mentioned above, can overcome a retarding voltage and reach the collector. A resolution of about 6% with a 5% transmission of the desired ions has been achieved.

It has been shown[19] that the resolution of the Bennett spectrometer may be substantially improved by altering the shape of the

applied r.f. voltage pulse. Townsend[20] has constructed a compact model of this instrument for use, while rocketborne, in investigating the composition of the upper atmosphere. Analysis and evaluations of energy-gain mass spectrometers have been given by Kerr.[21]

Cyclotron-resonance instruments possess high resolution and are designed primarily for the study of atomic masses. The Omegatron[22] is essentially a small cyclotron in which ions are accelerated if the frequency of the r.f. accelerating field coincides with the cyclotron frequency. The r.f. field is both uniform and weak, prevading the entire accelerating region, rather than possessing the dee established shape characteristic of high energy accelerators. Ions are created in the central region by electron bombardment, and spiral out to the collector. They are prevented by a d.c. field from escaping axially. A large number of revolutions results in a sharp mass discrimination, corresponding to a resolution of 1/10,000 for low masses. The mass spectrum is obtained by varying either the frequency or the magnetic field.

Consideration of these characteristic designs of mass spectrometers in view of present state of the art of electronics miniaturization indicates that an apparatus of this nature could be fabricated in to a very compact package. Fortunately recent development of miniature atmosphere analyzers can indicate what characteristics we may expect from a

miniaturized mass spectrometer leak detector.

Miniaturized Mass Spectrometer

An atmosphere composition analyzer for use in satellites has been developed by G. J. O'Halloran [23, 24] and patterned after the Bendix time-of-flight mass spectrometer. The basic advantages that can be gained by using this type of mass spectrometer stem from its inherent simplicity, versatility, and ruggedness and its unusually high speed of analysis and high signal to noise ratio. The success of the Bendix time-of-flight instrument resulted directly from the application of two inventions made at the Bendix Research Laboratories. The first of these was the development of the double grid source[25] which focuses all peaks, regardless of mass, with but one setting of the instrument operating parameters. The other is the development of a unique ion detector[26] of simple and rugged construction, which has an unusually high gain and extremely wide band pass characteristics.

The focusing action of the source is such that the time interval during which a particular mass peak is detected is much shorter than the time interval during which the original ionization took place. This makes possible a very substantial increase in the signal-to-noise ratio over that obtainable in instruments which utilize continuous ionization and detection, and substantially greater resolving power than has heretofore been available in time-of-flight mass spectrometers. The

new ion detector is a magnetic electron multiplier which employs crossed magnetic and electric fields to control the electron trajectories. The numerous dynodes of the more conventional multipliers have been replaced with a "continuous dynode" and a "field forming" strip, each consisting of a high resistance coating which has been fired onto an insulating support. This eliminates the need for special cathode and dynode contours and allows a straightforward mechanical design of the gating section mounted at the output end of the multiplier. The essential features of the Bendix time-of-flight mass spectrometer are shown in Fig. 15. The gas to be analyzed enters on the extreme left of the figure and is ionized by a pulsed electron beam. The resulting group of positive ions is ejected from the source region by another electrical pulse which is applied to the accelerating grids. These grids are arranged such that all ions which leave the source do so with the same energy. Consequently, the ions have a velocity distribution which depends on their mass-to-charge ratio. In this way, the ions separate into bunches such that each bunch contains ions of only one mass, and the separation becomes larger as the entire group proceeds down the separating region.

As the individual packets of ions arrive at the end of the separating region, they collide with the cathode of the magnetic electron multiplier. Each collision produces a packet of secondary electrons such that there

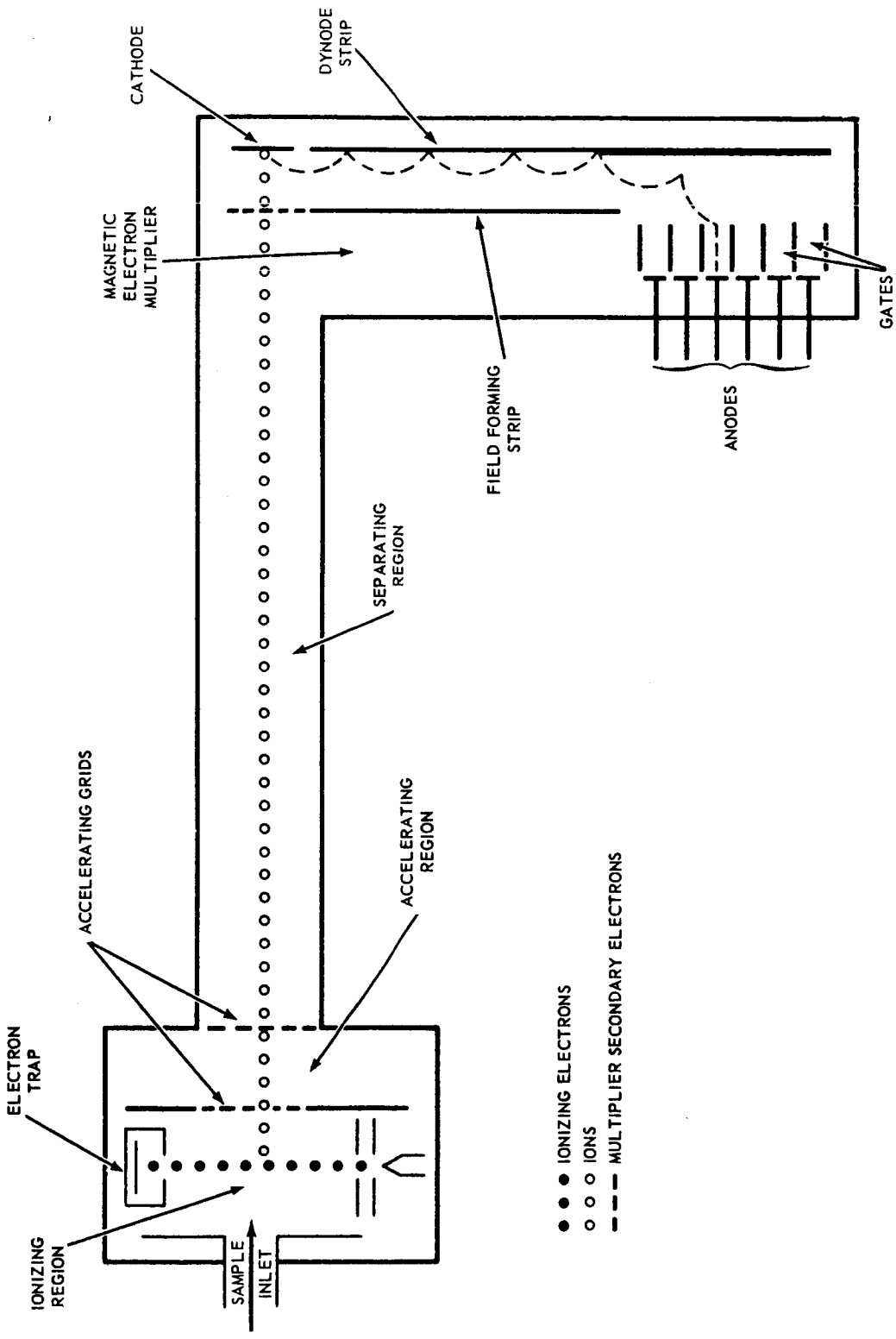

FIGURE 15 - Essential Features of the Bendix Time-of-Flight Mass Spectrometer

is a quantitative correspondence between the number of secondary electrons and the number of arriving positive ions. Since the magnetic electron multiplier is of the crossed magnetic and electric field design, the electron packets cycloid down the dynode strips in such a way that a current gain on the order of 10^6 is achieved by the time the packets enter the gating section of the multiplier.

The first packet of electrons, which corresponds to the sample constitutent of lowest mass, arrives at the entrance of the gating section about 2 microseconds after the group of ions leave the source. The last packet, corresponding to the heaviest sample constituent, arrives in 15 to 20 microseconds. In this way, the gating section of the multiplier is supplied with a series of electron packets which are spread out in time over an approximately 20-microsecond interval. This whole process of mass separation is repeated at a rate between 10 and 100 kilocycles per second so that a new group of molecules is being ionized and separated by mass every 100 microseconds or less.

The gating section of the magnetic electron multiplier can be fitted with up to 10 or more pairs of gate anode assemblies (6 are shown in Fig. 15). These gate-anode pairs have a configuration such that an electrical pulse applied to the gate will cause the electron packet then passing the assembly, to be gated onto the corresponding anode. The electrical capacity of these gate-anode assemblies is sufficiently low

that extremely short (50-nanosecond) pulses can be applied to any gate, thereby allowing each anode to collect the electron current due to a particular mass in the sample. In this way, as many gases can be continuously monitored as there are gate-anode assemblies in the gating section.

Complete mass spectra, at the repetition rate of the instrument, can be obtained by continuously biasing a gate electrode and using appropriate display technique. Complete mass spectra can also be obtained by applying the gating pulse a little later in each operating cycle of the instrument. This procedure will produce a rapid scan of the entire mass spectrum to provide a complete mass analysis while using only one output channel. Continuous and simultaneous monitoring of several mass peaks can be accomplished by applying to the appropriate gate an electrical pulse at the proper time in each operating cycle of the instrument.

The Model 17-210 time-of-flight mass spectrometer incorporates these techniques to provide five channels which are capable of continuously and simultaneously monitoring five of the components of the breathable atmosphere, and a sixth channel which scans the remainder of the mass spectrum searching for unexpected components, or components not important enough to be assigned to a continuously-monitored channel.

The gated electron current is measured by precision electrometers. Well-designed, unity feedback circuitry with high open-loop gain is used to provide low drift and to preserve the required fast response characteristics. The electrometer output can take any of several forms but is usually provided in the standard 0 to 5 volts dc configuration suitable for most any recorder, computer, or control equipment.

In order to provide the versatility required in the many experiments which have been designed around the Model 17-210, the instrument contains sufficient pumping and sample handling capability to provide three complete modes of operation, as follows:

(1) For use where 2 inches or less distance is permissible between the sample inlet and the mass spectrometer. In this mode of operation, the instrument has negligible sample transit time. The sampling rate is approximately 5 to 20 milliliters per minute and the response time for 0 to 90 per cent response is 60 milliseconds.

(2) For use where about 3 feet is required between the sample inlet and the mass spectrometer. In this mode of operation, the instrument has sample transit time of approximately 0.1 seconds. The sampling rate is 5 to 100 milliliters per minute and the response time for 0 to 90 per cent response is 60 milliseconds. This system is designed for use with an

approximately 3-foot length of 0.01 inch stainless steel hypodermic tubing.

(3) For use where 25 feet is required between the sample inlet and the mass spectrometer. In this mode of operation, the instrument has a sample transit time of approximately 0.5 seconds. The sampling rate is approximately 1 liter per minute, and the response time for 0 to 90 per cent response is 60 milliseconds. This system is designed for use with an approximately 0.10 inch I.D. tygon tubing.

It will be noted that the response time as used in the above description is that length of time required for the output voltage from each channel to cover 90 per cent of full scale for a step-function change of partial pressure in the spectrometer source (as reckoned from the instant at which the output begins to change). Using this definition, the response time is less than 0.06 seconds for each of the three sample system configurations. The dynamic range of the instrument is at least 10^4 to 1 at the 28 AMU (nitrogen) position in the mass spectrum and it is appreciably greater at most other mass positions -- if longer response times times than those listed above are allowed.

The principles of this spectrometer have been incorporated in the miniaturized instrument for use in earth satellites. This instrument measures the particle pressure of 6 gases simultaneously and it is programmed such that it measures the partial pressure of 14 different

neutral and ionized sample constitutents during the operating cycle of the complete mass spectrometer system. This instrument has a dynamic range of over one million and has been designed to record these signal levels on magnetic tape for later transmission to a ground receiving station. The unit is entirely transistorized except for the output electrometer circuit and high voltage regulator tubes.

The weight of the entire unit is approximately 29 pounds and it consumes 69 watts of electrical power and is capable of providing 10 continuous output channels in addition to 2 scanning channels. This includes the pumping equipment suitable for maintaining adequately low pressures within the vacuum analyzer and for transporting the gas sample from remote regions of the satellite. The volume of the instrument is of the order of 1/2 cubic foot and it is emphasized that this design is by no means the ultimate in miniaturization or power reduction. It is noted that by easing some of the more difficult to satisfy constraints, the size, weight, and power requirements noted above can each be reduced by a factor of two.

With regard to the vacuum pumping associated with a portable instrument such as this a getter-ion type pump should be strongly considered. Among its attractive features are: no continuous fore-pumping required; relatively portable; can be operated in any position; fundamentally simple, no heaters, hot filaments or moving parts;

operable from 10^{-10} to 10^{-1} mm of Hg; abrupt exposure to atmosphere usually causes no damage; and power consumed by pump at low pressure is small; hence special cooling is not necessary.

The commercial Vac Ion pump of this type operates by taking gas molecules and atoms out of circulation by the formation of chemically stable compounds and by ion burial. This is achieved by three inter-related phenomena: ionization, sputtering, and chemical combination. The major components of the system are power supply, permanent magnet, and pump. The pump consists of an enclosure containing an anode grid sandwiched between two cathode plates.

Pumping is initiated by a suitable voltage between anode grid and cathode plates resulting in a cold cathode electron discharge. Electrons, tending to flow to the anode, are forced into a spiral path by a strong magnetic field. The greatly increased electron path length results in a high probability of collision between free electrons and gas molecules. These collisions produce gas ions and more free electrons.

These positively charged gas ions then bombard the titanium cathode plates and titanium atoms are knocked out of the plate (sputtered). The sputtered titanium atoms are deposited on the anode grid, forming chemically stable compounds with the active gas atoms such as oxygen and nitrogen. Chemically inert gases are also removed by ion burial and by entrapment on the anode.

Because each collision produces an increasing number of electrons with long effective path lengths, pumping action is maintained down to very low pressures (past 10^{-9} mm Hg).

Self regulation of titanium consumption is achieved because sputtering rate is virtually a linear function of the number of electron-atom collusions.

The system has to be rough pumped to approximately 10^{-2} mm Hg before activating the Vac Ion pump. This can be done by a conventional fore pump. Another means is by chilling with liquid helium[27] on appendage attached to the system. A related system employs activated charcoal chilled with liquid nitrogen[28] and is used in the commercially available Vac Sorb Pump.

Conclusion

The use of a mass spectrometer is the most sensitive method of detecting leaks and helium is an ideal tracer gas. As a result of the pumping system associated with the mass spectrometer and the high diffusion rate of helium it is possible to detect leaks via a "sniffer" at a relatively large distance from the instrument. A logical procedure would be to start at the top, anchor the instrument, check all the joints in the area by means of the sniffer tube, then move down as the helium diffuses upward. It may be advantageous at times to use different tracers in the various systems and detect them with a mass spectrometer.

This would reduce the background associated with any one tracer gas. By a balancing technique a mass spectrometer can easily be made a differential detector if that should prove more efficient.

The main disadvantage with a time-of-flight instrument is the limited resolution at the higher masses but this is not detrimental for leak detection purposes. A time-of-flight spectrometer has the advantage that its accuracy depends on electronic circuits rather than extremely accurate mechanical alignment and the production of highly uniform, stable magnetic fields. The freedom from stringent geometric conditions simplifies construction.

In this instrument, due to the pulsed beam, the ion formation process has a rather low duty cycle, commonly of the order of 0.1 to 10%, which would indicate that the ion currents would be much lower in intensity than those obtained with a conventional deflection type mass spectrometer. However the absence of narrow slits in the ion source and detector allows one dimension of the ion beam to be much larger than is possible in a conventional instrument. This allows a more effective utilization of any given electron beam in producing useful ions.

The theory as well as the results of development work associated with a miniaturized mass spectrometer of this type indicate that the ability and stability this instrument possesses will contribute greatly to the efficiency and quality of leak detection associated with missiles.

Proposed Bendix Time-of-Flight Mass Spectrometer

The proposed Bendix time-of-flight mass spectrometer as described by Dr. G. J. O'Halloran, Head of the Molecular Physics Department, Bendix Corporation, Southfield, Michigan, in a letter written to Ohio University on 29 November 1962.

Sensitivity - 2×10^{-3} SCIM at 1% Helium tracer and 1% pickup efficiency.

(Converted from 5×10^{-4} cc/sec)

	With Pump	Without Pump
Weight	10 lbs.	5 lbs.
Volume	415 cu. in.	145 cu. in.
Power	46.4 watts	6.4 watts

With hydrogen tracer total size and power are less.

Estimated cost -- $100,000 - $165,000.

Development time - 10 months.

Cost in lots of 100 - $4,000 each.

Features of Time-of-Flight Mass Spectrometer:

Vac-ion pump (no moving parts)
Magnetic electron multiplier
Time of flight mass determination

Sensitivity Comparisons:

Halogen detector - 10^{-2} SCIM.
Thermistor bridge - 10^{-1} SCIM.

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CHAPTER VII

ACOUSTICAL METHODS

Ultrasonic Leak Detection Study

On first analysis it seems that several factors should influence the character of noise produced by gas escaping through a leak. These are:

1. Pressure difference.
2. Leak geometry.
3. Nature of the leaking gas.

There are also at least three mechanisms which might be responsible for the noise:

1. Vortices formed at the sharp inner edge of the orifice and expelled into the air at the outer edge.
2. Turbulence caused by the high velocity flow in the orifice itself (comparable to turbulence in a tube).
3. Turbulence caused in the surrounding air by the stream or jet moving out into the stable air.

The first of these mechanisms would be expected to produce a sharply peaked spectrum of noise since this leads to a frequency given by

$$f = AU/L$$ where A = a constant
U = fluid velocity
L = thickness of orifice.

This frequency is simply the rate at which vortices would reach an observer (i.e., velocity of vortices/distance between vortices). In this argument it is assumed that the creation length of a vortex is proportional to L.

Flow through the orifice may be turbulent since the Reynolds Number for a typical leak at 2,000 psi can be shown to be much greater than 3,000 at which value turbulence begins even in a smooth pipe. Noise due to turbulence in the leak itself would depend on hole geometry.

Sound produced by the third mechanism is broadband in character. Mawardi and Dyer [1, 2] have worked out general noise spectrum curves for all jet noises (air jets and jet engine turbulence). These curves are presented in Fig. 16.

An experimental study of leak noise spectra has been carried out in this laboratory to obtain data for use in optimization of the leak detection method based on this phenomenon. The laboratory setup is shown in Fig. 17. Equipment used included:

Microphone...Bruel & Kjaer Type 4135, serial No. 77219.

Amplifier...Bruel & Khaer Type 2604, serial No. 82452.

Spectrum analyzer...Probescope Model SS-500.

Camera...Hewlett Packard Model 196A.

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[1]Superscript numerals refer to references listed at the end of this chapter.

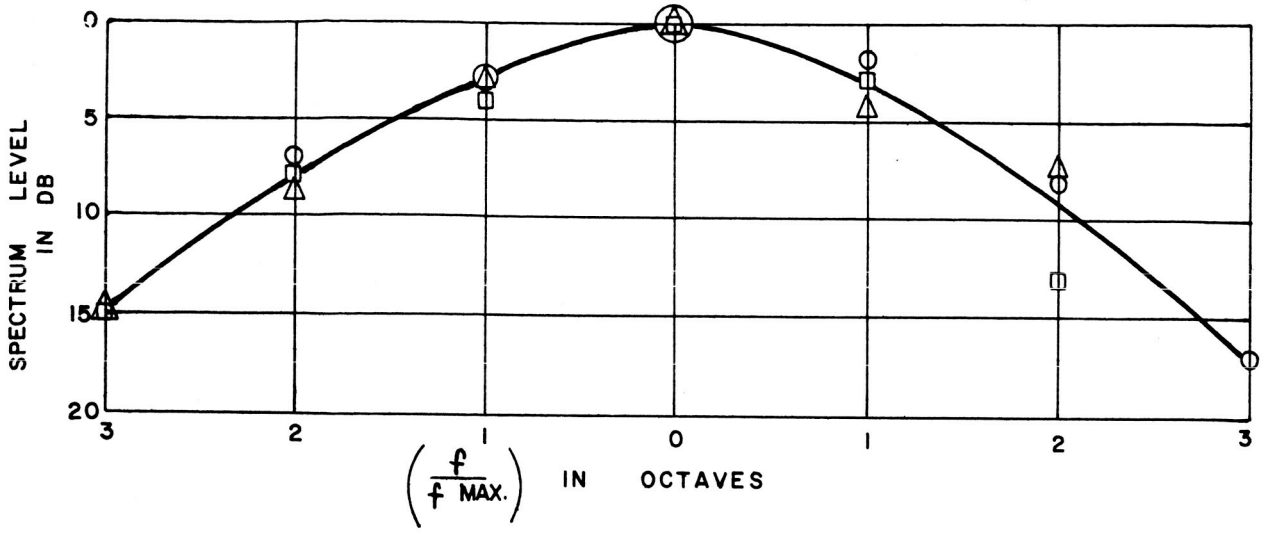


FIGURE 16

RELATIVE SOUND LEVEL VS FREQUENCY

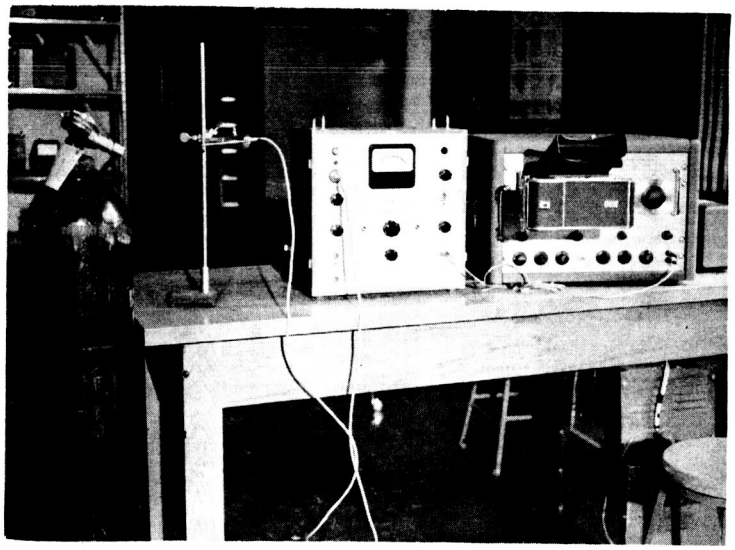


FIGURE 17

EQUIPMENT USED IN ULTRASONIC STUDY

The microphone was furnished with a calibration curve that showed it to be reasonably flat from 50 cycles per second to 100 kilocycles per second. The linearity of the amplifier and spectrum analyzer was tested by feeding white noise into the amplifier and checking the picture on the screen. The linearity of the system proved quite satisfactory.

Leaks on both high and low pressure systems were used in the tests. Low pressure varied from 1 to 70 psi, while high pressure ranged from 500 to 2,000 psi.

Leaks used at low pressure were calibrated round holes as well as diffuse leaks produced by loosening flange nuts. High pressure leaks were calibrated holes of about 0.001 inch diameter.

At low pressure the leaking gas was air, while at a high pressure nitrogen and helium were used.

While the leaks varied considerably in amount of acoustical energy produced there was little variation in the spectral distribution of this energy. As the figures show (Fig. 18 to Fig. 53), all leaks had spectra peaked broadly at a frequency between 35 KCPS and 45 KCPS. Falloff at one octave below this range was 10 db. At one octave above the spectrum was down 20 db.

Table IV lists experimental data pertinent to the figures.

These experiments lead to the conclusion that the third mechanism

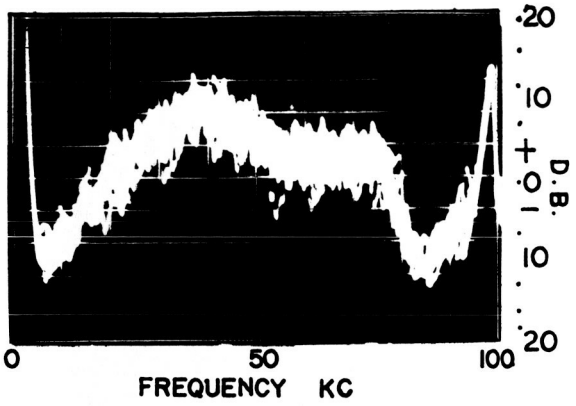


FIG 18

70 PSI AIR 40 SCIM

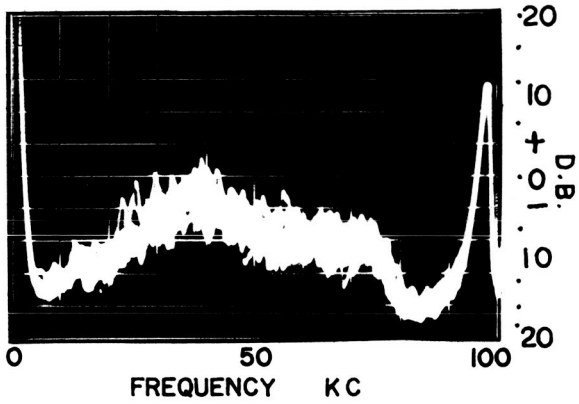


FIG 19

30 PSI AIR 19.5 SCIM

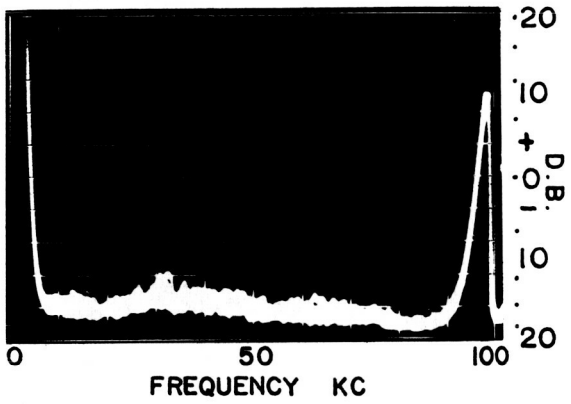


FIG 20

10 PSI AIR 9 SCIM

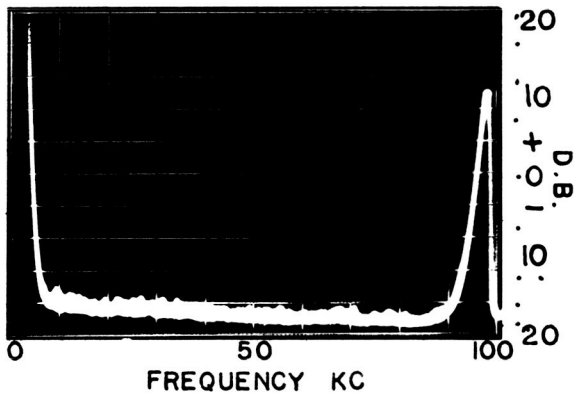


FIG 21

0 PSI AIR 0 SCIM

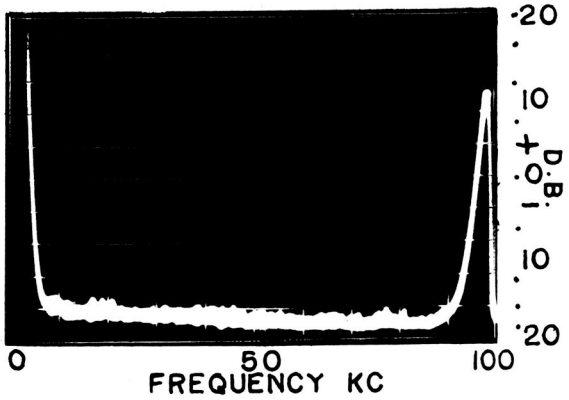


FIG 22

80 PSI AIR 4.8 SCIM

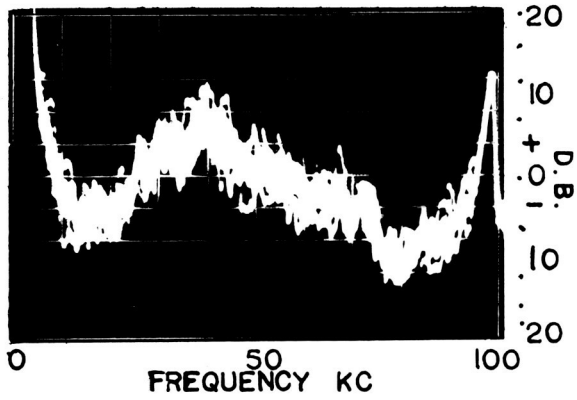


FIG 23

10 PSI AIR 9 SCIM

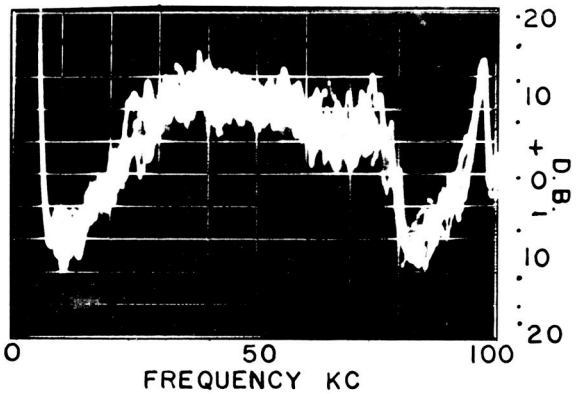


FIG 24

70 PSI AIR 40 SCIM

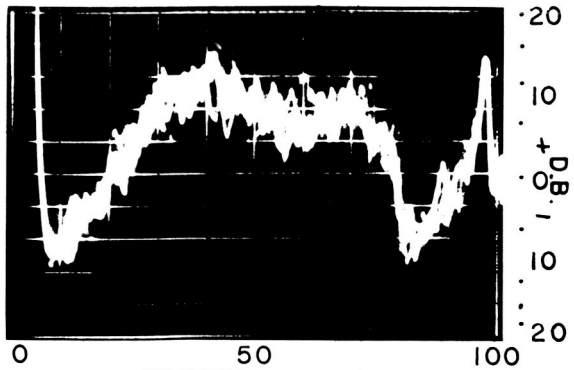


FIG 25

70 PSI AIR 40 SCIM

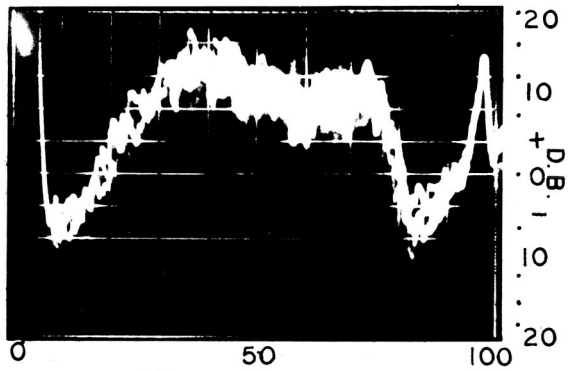


FIG 26

70 PSI AIR 40 SCIM

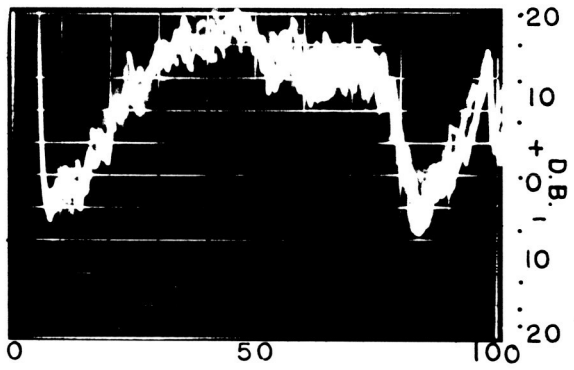


FIG 27

70 PSI AIR 40 SCIM

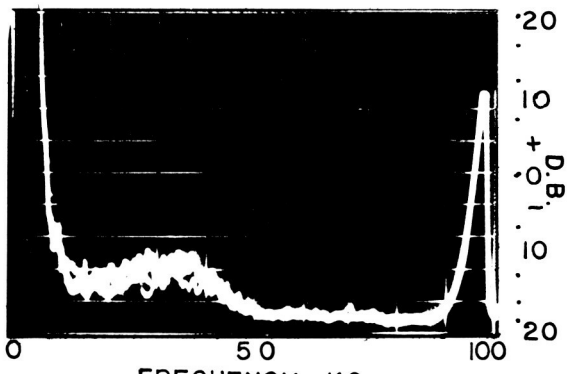


FIG 28

70 PSI AIR 40 SCIM

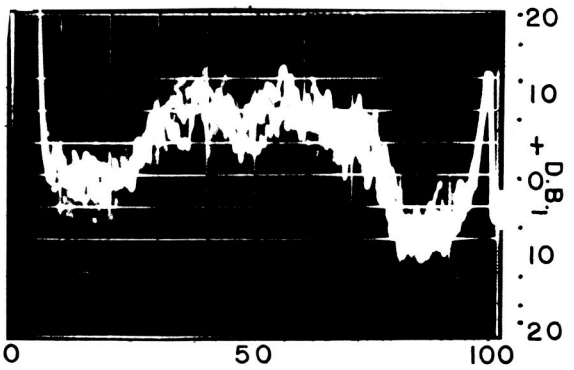


FIG 29

90 AIR

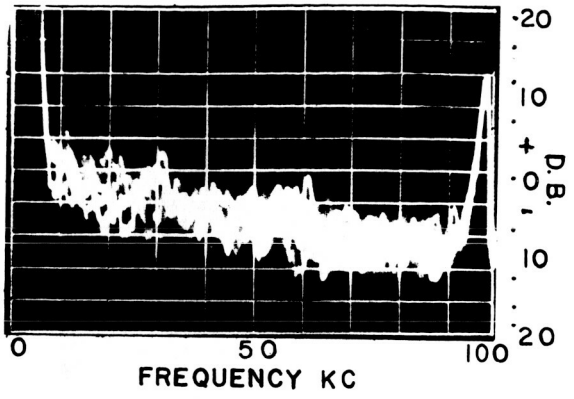


FIG 30
BACKGROUND NOISE

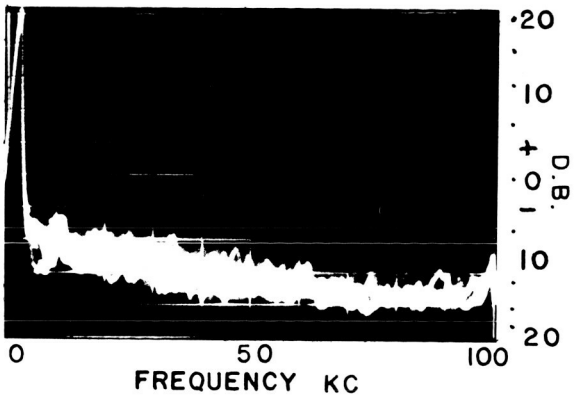


FIG 31
BACKGROUND NOISE

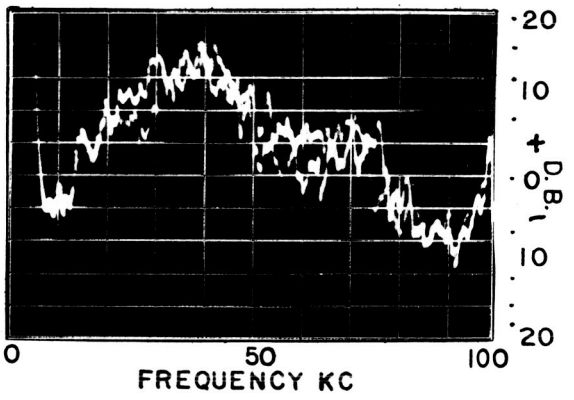


FIG 32
500 PSI He 50 SCIM

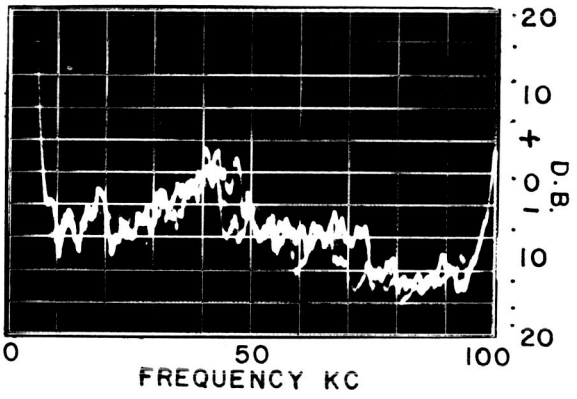


FIG 33
500 PSI N_2 25 SCIM

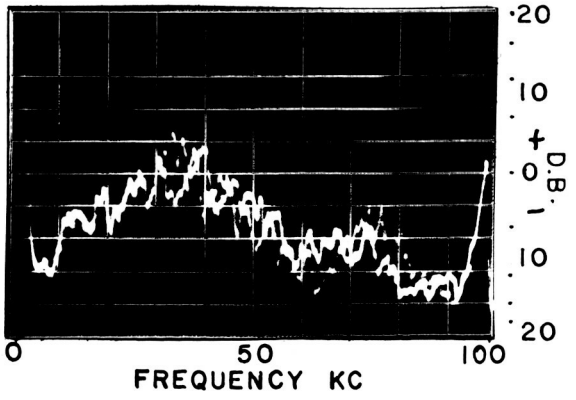


FIG 34
1000 PSI He 120 SCIM

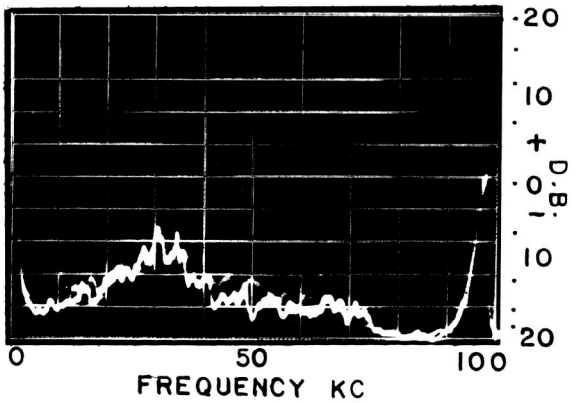


FIG 35
1000 PSI N_2 60 SCIM

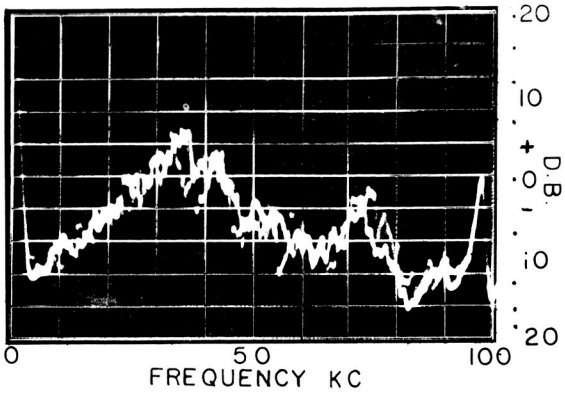


FIG 36

2000 PSI H_e 250 SCIM

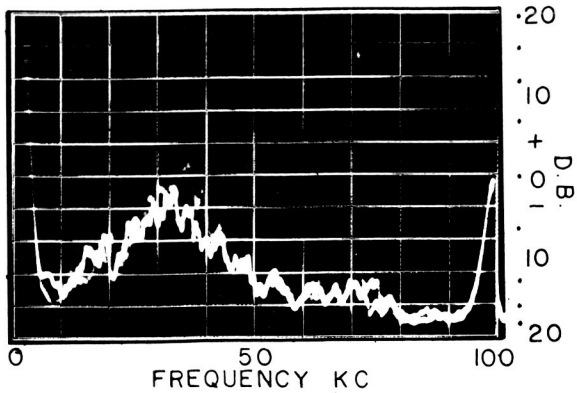


FIG 37

2000 PSI N_2 130 SCIM

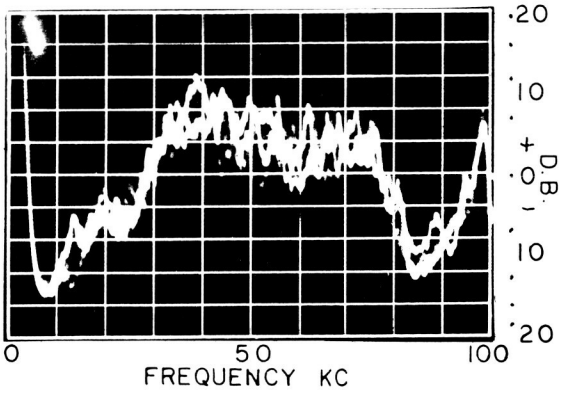


FIG 38

2000 PSI N_2 130 SCIM

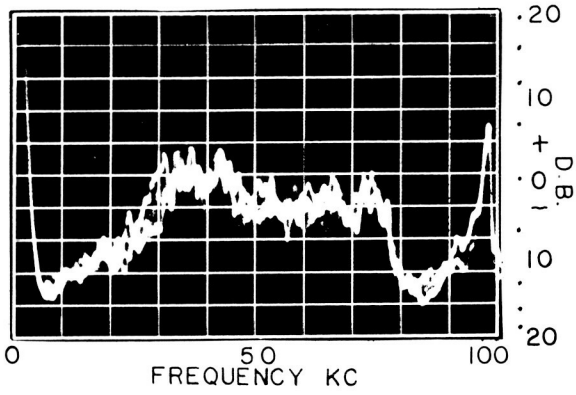


FIG 39

2000 PSI N_2 130 SCIM

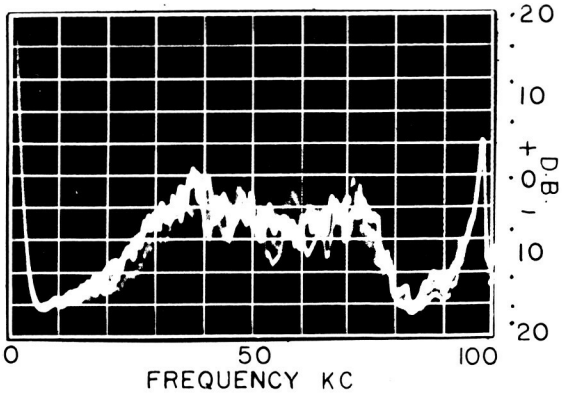


FIG 40

2000 PSI N_2 130 SCIM

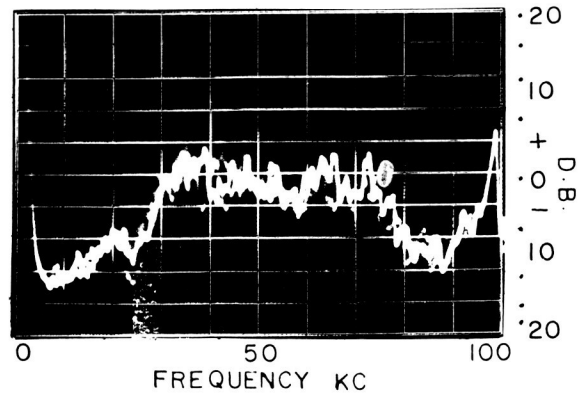


FIG 41

1000 PSI N_2 60 SCIM

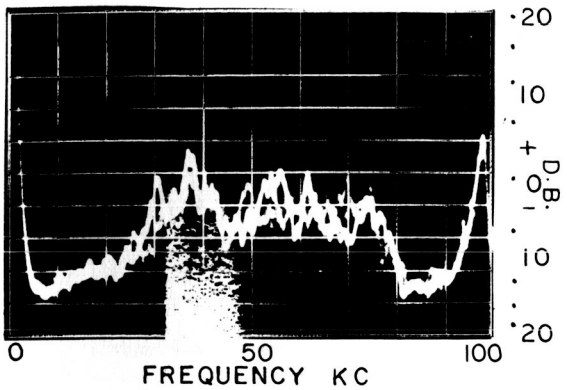


FIG 42

1000 PSI N_2 60 SCIM

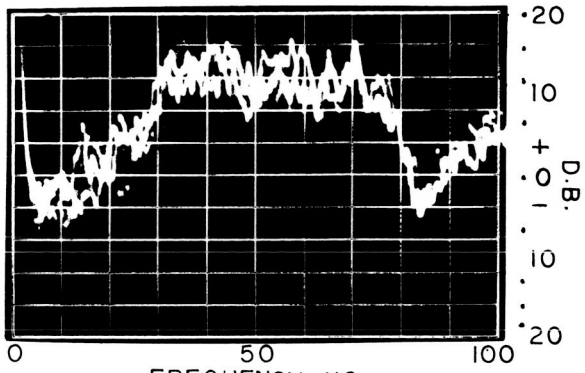


FIG 43

2000 PSI N_2 130 SCIM

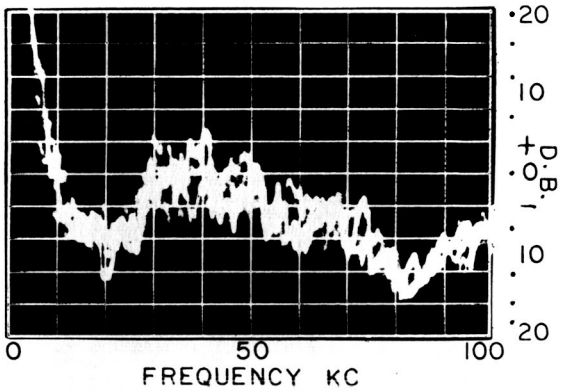


FIG 44

1950 PSI N_2 3 SCIM

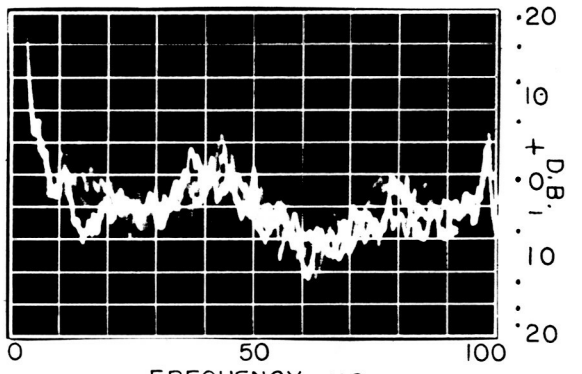


FIG 45

2000 PSI N_2 3 SCIM

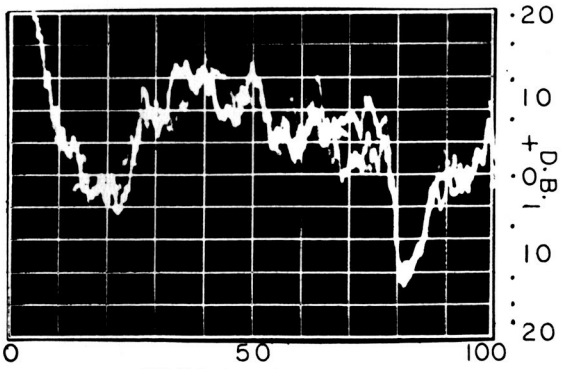


FIG 46

2000 PSI H_e 7 SCIM

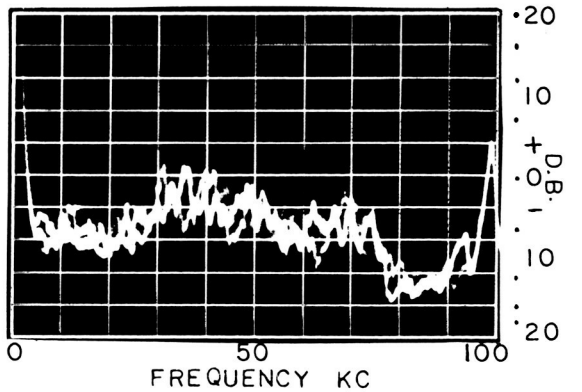


FIG 47

2000 PSI H_e 7 SCIM

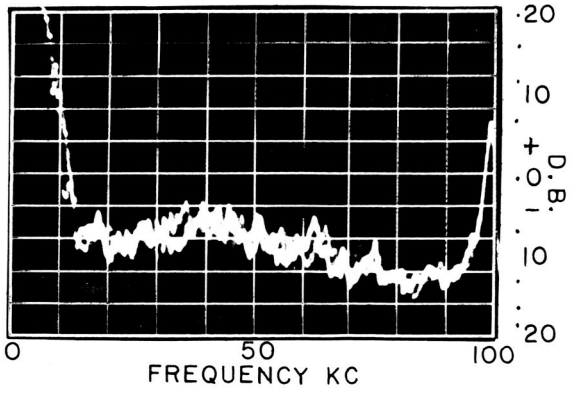


FIG 48

2000 PSI N_2 3 SCIM

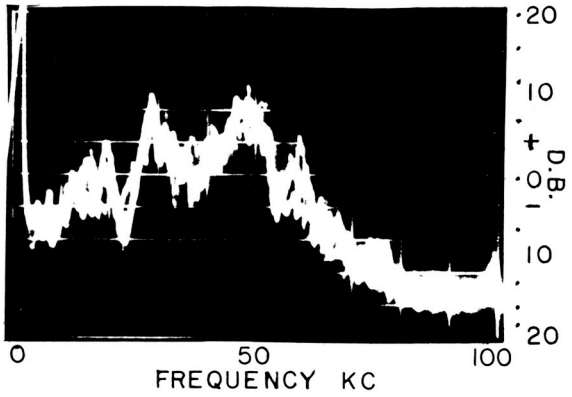


FIG 49

2000 PSI N_2

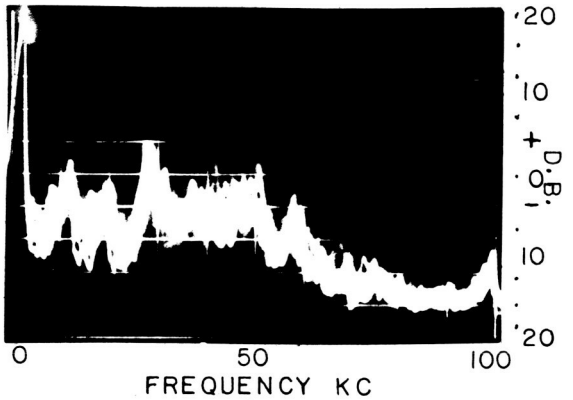


FIG 50

1500 PSI N_2

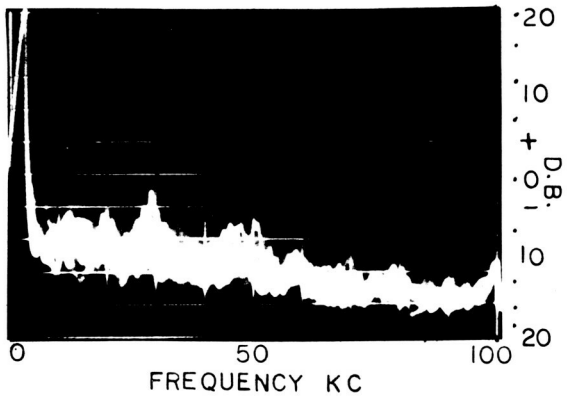


FIG 51

1500 N_2

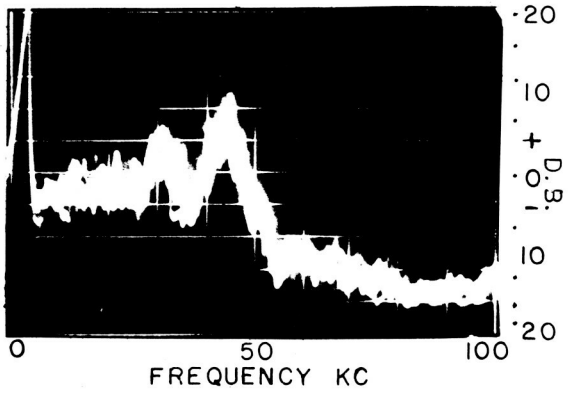


FIG 52

1500 PSI N_2

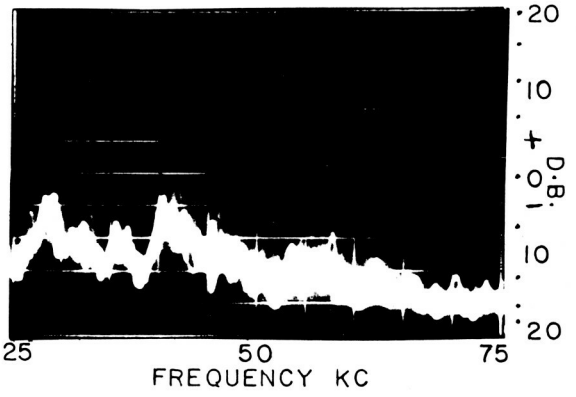


FIG 53

2000 PSI N_2

TABLE IV

Fig.	Leak	PSI	Gas	SCIM	Odb = ubars	Dist. from Leak	Remarks
18	A	70	air	40	1.5	10 cm.	at 45°
19	A	30	air	19.5	1.5	10 cm.	45°
20	A	10	air	9	1.5	10 cm.	45°
21	A	0	air	0	1.5	10 cm.	45°
22	B	80	air	4.8	1.5	10 cm.	45°
23	A	10	air	9	1.5	1 cm.	45°
24	A	70	air	40	1.5	10 cm.	45°
25	A	70	air	40	1.5	10 cm.	45°
26	A	70	air	40	1.5	10 cm.	45°
27	A	70	air	40	1.5	10 cm.	30°
28	A	70	air	40	1.5	10 cm.	0°
29	Diff	90	air	--	--	--	45° 90-45#/65 sec
30	Background noise						
31	Background noise						
32	D	500	He	50	0.3	35 cm.	5°
33	D	500	N_2	25	0.3	35 cm.	5°
34	D	1000	He	120	1.0	35 cm.	5°
35	D	1000	N_2	60	1.0	35 cm.	5°
36	D	2000	He	250	1.0	50 cm.	5°
37	D	2000	N_2	130	1.0	50 cm.	5°
38	D	2000	N_2	130	1.0	25 cm.	5°
39	D	2000	N_2	130	1.0	50 cm.	5°
40	D	2000	N_2	130	3.0	20 cm.	5°
41	D	1000	N_2	60	0.3	25 cm.	5°
42	D	1000	N_2	60	3.0	25 cm.	5°
43	D	2000	N_2	130	0.4	20 cm.	5°
44	E	1950	N_2	3	0.05	1.2 cm.	5°
45	E	2000	N_2	3	0.2	0.5 cm.	5°
46	E	2000	He	7	0.4	1.0 cm.	5°
47	E	2000	He	7	0.4	4.0 cm.	5°
48	E	2000	N_2	3	0.4	1.0 cm.	5°
49	F	2000	N_2	--	0.4	10 cm.	5°
50	F	1500	N_2	--	0.4	10 cm.	5°
51	F	1500	N_2	--	0.4	10 cm.	5°
52	F	1500	N_2	--	0.4	2 cm.	5°
53	F	2000	N_2	--	0.4	2 cm.	5° 25-75 KC

Leak A shown in Fig. 55 and Fig. 56.
Leak B shown in Fig. 55 and Fig. 56.
Leak D and E are small holes in high pressure caps.
Diffuse loose connection.
Leak F is a loose cap.

above is the significant one. They also indicate that the instrument tested, the Delcon Ultrasonic Translator, which has been designed to respond to this peak, should be an optimum design, frequency-wise. It may be possible to improve on its sensitivity (which was found wanting) by a redesign of the transducer to incorporate controllable directivity of the probe or variable bandwidth, but no such effort has been undertaken here.

Evaluation of the Ultrasonic Translator

1. Leak Detection Distance

Fig. 54 shows the small compressed air reservoir containing a leak plug and pressure gage, and the flow meter used for measuring the larger air flows. Smaller air flows were measured by the water displacement device shown in Fig. 55.

The leak plugs and flange also shown in Fig. 55 are described in Fig. 56.

The ultrasonic detector was put directly in front of the leak, on the axis of the cylinder, and the maximum detection distance at full volume setting noted. Distance vs. leak flow rate is shown in Fig. 57. This figure also illustrates that detection distance for a given flow rate varied with the individual leaks. The flange leak showed the greatest variation -- it was detectable only in the plane of the flange, not on the cylinder axis, and the detection distance was

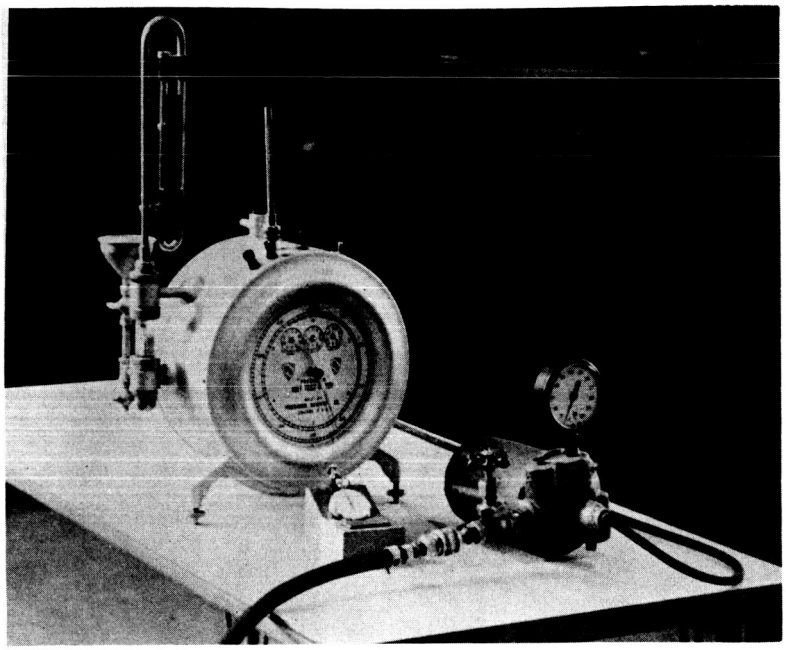


FIGURE 54

EXPERIMENTAL LEAK CHAMBER & FLOW METER

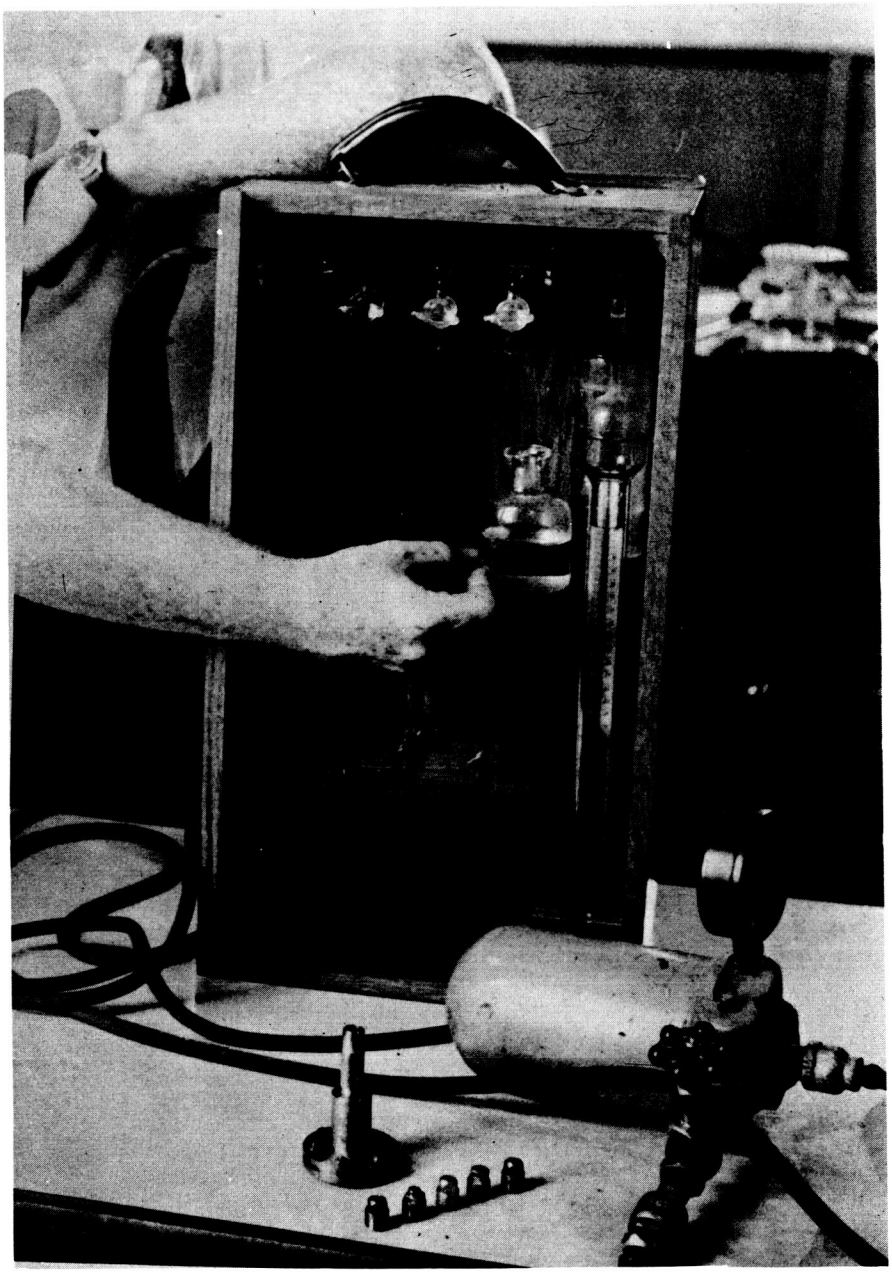
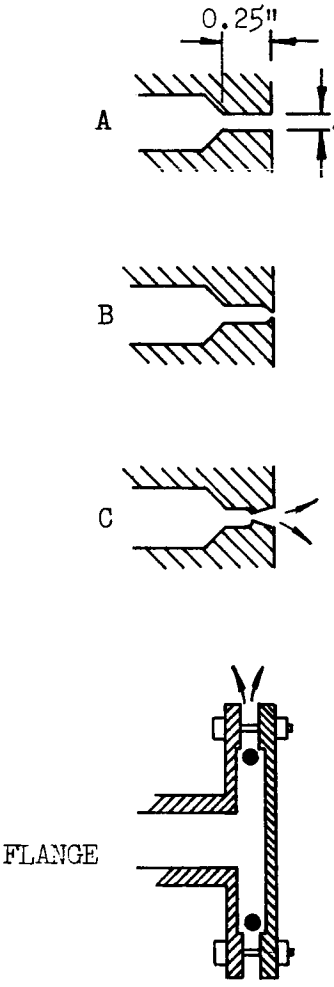


FIGURE 55

EXPERIMENTAL LEAK CHAMBER WITH LEAK
PLUGS AND WATER DISPLACEMENT DEVICE



LEAK	FLOW, SCIM		PRESSURE, PSIG	
	Min.	Max.	Min.	Max.
A	3.4	42.8	2	75
B	1.01	4.27	25	75
C	4.3	18.3	5	50
FLANGE		25		15

FIGURE 56

LEAKS

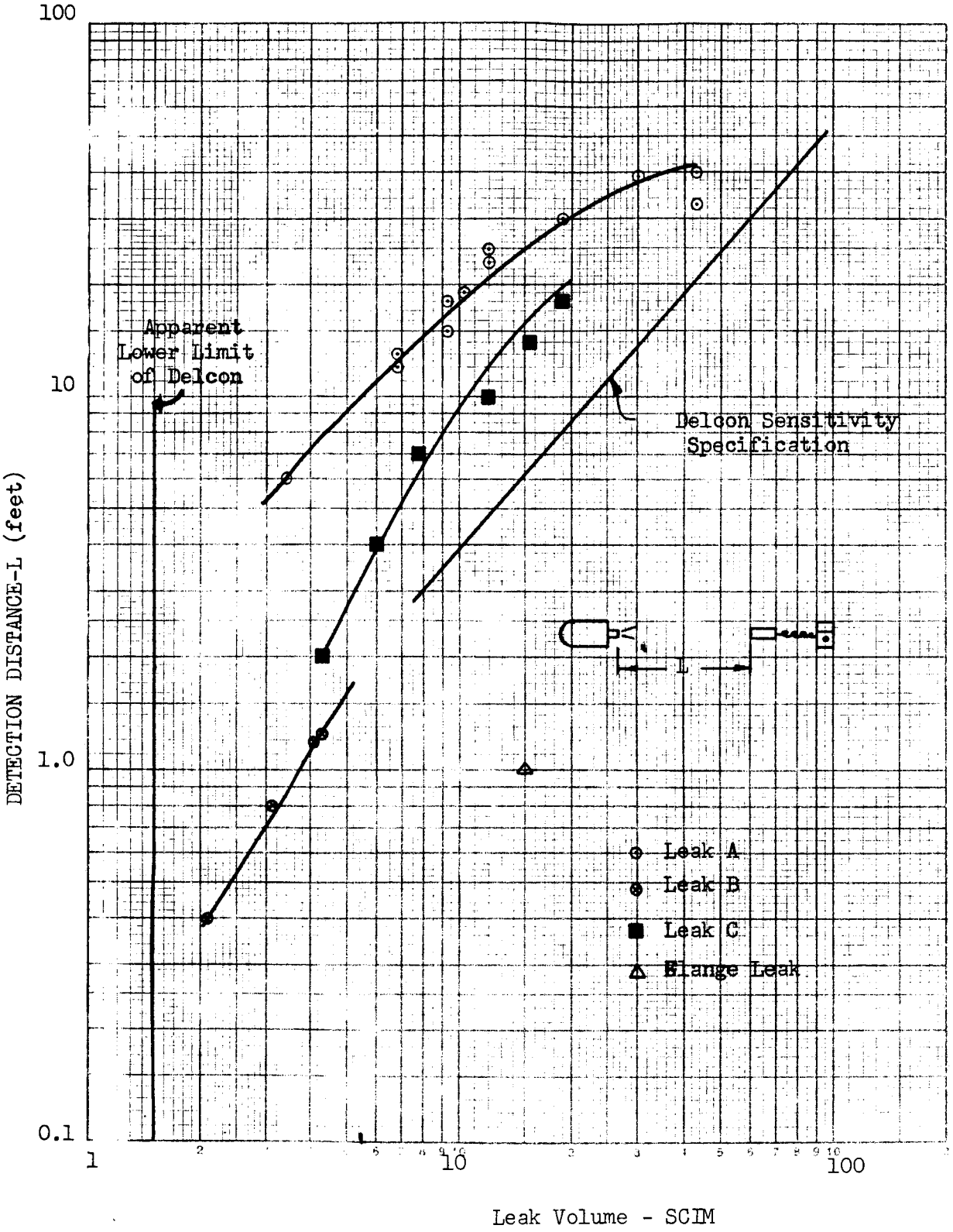


FIGURE 57

DELCON ULTRASONIC LEAK DETECTOR
DETECTION DISTANCE VS LEAK FLOW RATE

reduced as might be anticipated from the diffuse flow of the leak.

Wetting the leaks with a soap solution produced variations in detection distance from zero to two or three times dry value.

On some of the leaks it was noted that the human ear was as sensitive as the ultrasonic detector, but the detector was always superior in high environment noise conditions.

2. Effect of Small Obstruction between Leak and Detector

The detector probe was moved in a horizontal plane around the leak tank with the effect on detection distance shown in Fig. 58. At each point on Fig. 58, the detector probe was pointed so as to maximize the signal.

3. Effect of Environmental Noise

The ultrasonic leak detector was sufficiently directional to be unaffected by the environmental noise present in the test described above; namely, a one-horsepower electric motor driven air compressor and metal tools pounding on metal about 40 to 90 degrees and 15 feet away from the detector. The detector would pick up large signals from these sources when pointed directly at them. It was estimated that a 15^{o} deviation of the detector probe from such noise sources was sufficient to reduce their signal to essentially zero. But it must be pointed out that noise from a source far removed from the line of aim

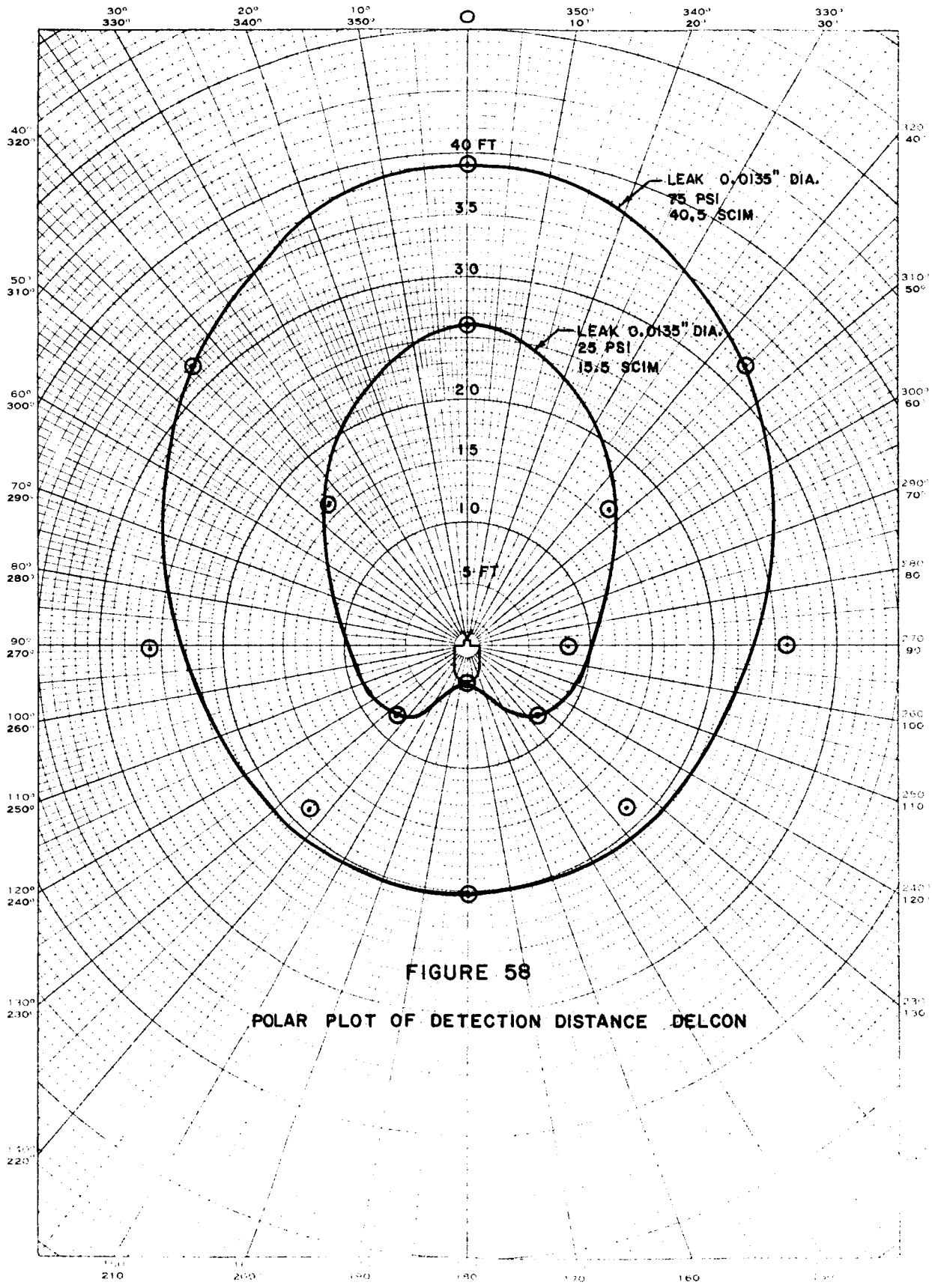


FIGURE 58

POLAR PLOT OF DETECTION DISTANCE DELCON

may still be received by reflection from walls, etc.

4. Conclusions and Remarks

The Delcon ultrasonic detector seems useful for quick detection of leaks above about 2 SCIM if they are not diffused, shielded by an obstruction, or otherwise hindered in their acoustic propagation characteristics.

In booster checkout work, this detector may be useful for locating and correcting larger leaks before charging the system with halogen tracer.

Sound Injection and Correlation Method

A method of leak detection currently being explored by the American Gas Association for use in locating leaks in buried gas lines seems worthy of consideration in connection with the present study. This method uses a transducer to inject into the gas in the pipe a 400 cycle per second sound wave. A pickup system is used to explore at the surface along the pipe for the leak which may be several feet underground. The signal injected in the pipe comes out with the leaking gas. A second oscillator generates another 400 cycle per second signal in the detector system which is combined with the signal from the leak in a crosscorrelation detector circuit. As the phase of the signal generated in the pickup circuit is varied, peaks are obtained

from the crosscorrelation function when the two signals are in phase. The largest crosscorrelation peak occurs when the injected signal is largest, which is where the pickup is directly over the leak.

The concept of leak pinpointing by means of a sonic tracer signal has been tested experimentally in a pilot facility located at the Institute of Gas Technology. This facility consists of a 2 inch steel main approximately 220 feet in length, buried to a depth in the ground varying from 2 to 5 feet. A section of this main is 4 1/2 feet below a 30 foot wide 4 inch concrete paving.

According to reports of the Gas Institute, 50 milliwatts of injected power was sufficient for pinpointing a 0.125 inch diameter orifice under the concrete. The method is also reported to have been successful on a bell-and-spigot joint leak, and orifice type leaks from 0.050 to 0.125 inches in diameter located at various positions around the pipe.

There are manifest differences in the situation in gas line leaks underground and leaks in the Saturn missile system. The gas pipe has a covering of earth which is perhaps a good conductor of sound waves and which contributes to the vibrational stability of the system. The Saturn has a more complex geometry, a smaller critical leak size, and higher pressures. It would seem, however, that the sonic injection system offers enough promise to merit further study.

References:

1. Richardson, Technical Aspects of Sound, Elsevier Publishing Co., New York, N.Y. (1957), p. 373, Volume 2.

2. O. Mawardi and I. Dyer, J. Acoust. Soc. Am. 25, 389 (1953).

3. American Gas Association Research Project PB-36 Report DMC-61-22. Institute of Gas Technology, Chicago, Ill.

4. Mine Safety Appliances, 201 North Braddock Avenue, Pittsburgh 8, Pa., is developing this leak detector and hopes to have it out by the end of 1963.

5. Some Facts About the A.G.A. Research Program on Sonic Pinpointing of Gas Leaks. Undated release of the Institute of Gas Technology, Chicago, Ill.

CHAPTER VIII

SYSTEMS CONCEPTS - RADAR-CAVITY SYSTEM

General

Any problem as involved as that of leak detection and location in missile systems eventually must be solved from the systems viewpoint. This approach may well begin with a microwave communication system for gathering information concerning leaks and transmitting it to a control center for processing and subsequent action. Research teams working on this project have considered two such systems: one passive, one active. A passive system is here defined as one in which the control center supplies the energy for interrogation of the leak detectors, while an active system is considered to be one in which the energy for transmission of the information must be supplied by sources built into the detector-transducers. A further topic for investigation has been the nature of the transducers themselves.

Resonant Cavity Scanning System - the Passive Approach

The ultimate system of leak detection, as contrasted with a temporary or stop-gap measure, requires the following properties:

(a) The detector-responder device affixed to the missile at numerous locations must be minimum in weight, cost, and power requirements. On the other hand, a centrally located interrogator device could be more bulky weight-wise and higher in cost.

(b) A minimum effort should be required to obtain information from the detectors. If the number of detectors is great, a one-at-a-time connection done manually would be impractical, especially during the most critical periods such as countdown.

(c) Background noise and interference must be largely eliminated so far as the detection scheme is concerned.

(d) A positive identification system must be established whereby the test operator can rapidly determine the leak location, preferably without requiring a point-by-point check at close proximity.

(e) Due to the complexity of the missile structure, it is important that the detectors have ready accessibility.

A microwave system satisfies to a large extent all of the above requirements. Using X- or K-band frequencies, resonant cavities of small size and weight can be extended to critical locations on the missile, each tuned to a specific frequency of response, and actuated by a trigger device at the most likely location. The interrogator-responder device can be located a reasonable distance away from the detectors, and can be of reasonable size and cost. This device will scan the frequency band covered by the resonant cavities and will indicate to the operators those transponders which give a positive indication of a nearby leak. In the range of frequencies covered by X- and K-band, background noise offers little or no interference.

In remote or inaccessible locations, with a microwave system reasonable lengths of light-weight coaxial cable can be used to facilitate information transmission.

An X-band microwave system has been in operation on several occasions. The system consists of an APG-32X-band radar with a directional scan antenna, 30 kilowatts peak power, 9245 mc. operating frequency, one-half microsecond pulse width and a pulse repetition frequency of 2,000 cycles per second. With the cavity located 40 feet from the interrogator, a saturation response was received for a duration of 2.5 microseconds. A cylindrical resonant cavity 4.9 centimeters in diameter and 2.75 centimeters in length with a horn aperature 2.4 by 2.9 centimeters gave adequate response. Even with metallic obstructions located in the radar beam, little or no interference was noted in cavity response. Due to the long ringing time, compared to the one-half microsecond transmitted pulse, extraneous noise caused practically no interference. The highly directional nature of the APG-32 antenna enables it to pinpoint the transponder locations.

The good response obtained with a minimum-sized cavity may make it possible to use X-band components rather than the more expensive K-band units originally planned, provided that the total weight and number of detectors are not excessive. The research team has experimented with simplified couplings, horns, tuners, and

cavities which can be produced at lower cost and still give satisfactory ringing time with the ample interrogator power available.

For the more inaccessible leak locations, experimental data have been obtained for ringing times using various lengths of RG-58A/U coaxial cable. Using a horn with a mouth dimension of 4 by 3 inches and cable lengths up to 20 feet, return ringing times of from 1.5 to 2.5 microseconds were obtained which were ample for detection purposes.

In order to simulate actual conditions of use, no effort was made to minimize reflections from metal Venetian blinds, conduit pipes, steel-encased windows, and other irregularities. Fig. 59 indicates the conditions of test in this respect. These reflections, in addition to the 0.5 microsecond transmitter pulse, show up quite plainly in Fig. 60 and Fig. 62. The horns and cavities used were of commercial manufacture and are shown in Fig. 63 and Fig. 64.

The results of these experiments indicate that the smaller horn with an aperature of 2.4 x 2.9 cm. and an axial length of 7.5 cm. is entirely satisfactory. The response using these components is shown in Fig. 61. From this it is evident that the receiver is saturated for approximately 1.5 microseconds, excluding the transmitter pulse. To prevent saturation of the receiver, a 15 db attenuation in the IF strip was required.

The response, or ringing time, of a system of this type depends on its overall Q. This overall Q can be determined from the reciprocal

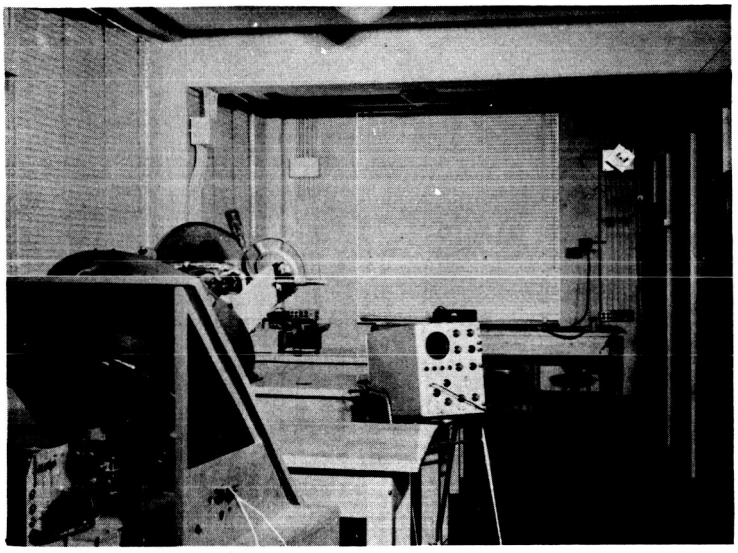


FIGURE 59

LABORATORY ARRANGEMENT FOR MICROWAVE SET-UP

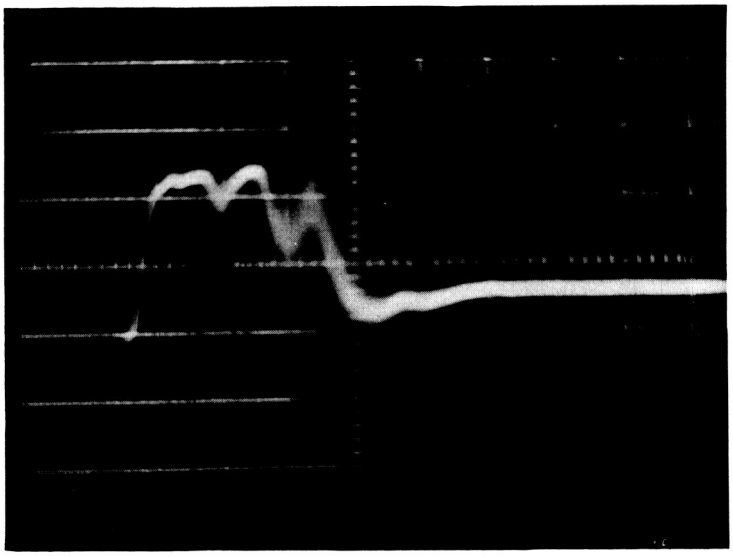


FIGURE 60

RADAR VIDEO PULSE CAVITY
TUNED TO 9240 MC

FIGURE 61

RADAR VIDEO PULSE CAVITY
TUNED FOR MAX RESPONSE

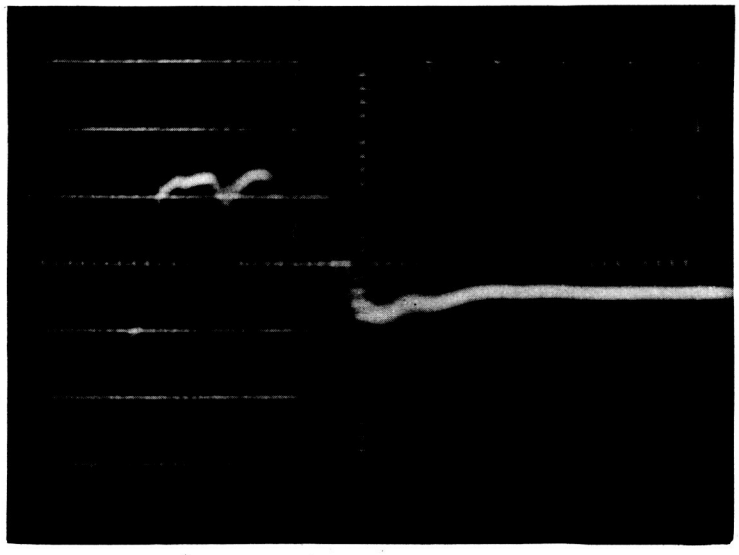


FIGURE 62

RADAR VIDEO PULSE CAVITY DETUNED
TO LOWER EDGE OF BAND

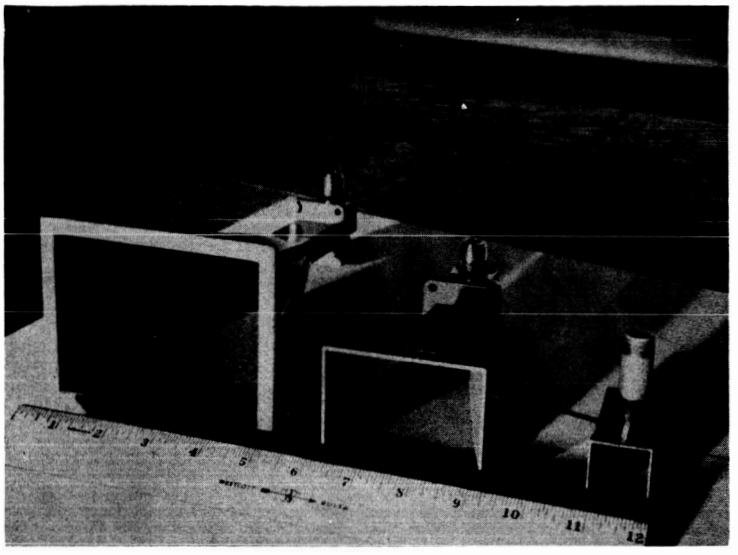


FIGURE 63

HORN CONFIGURATIONS

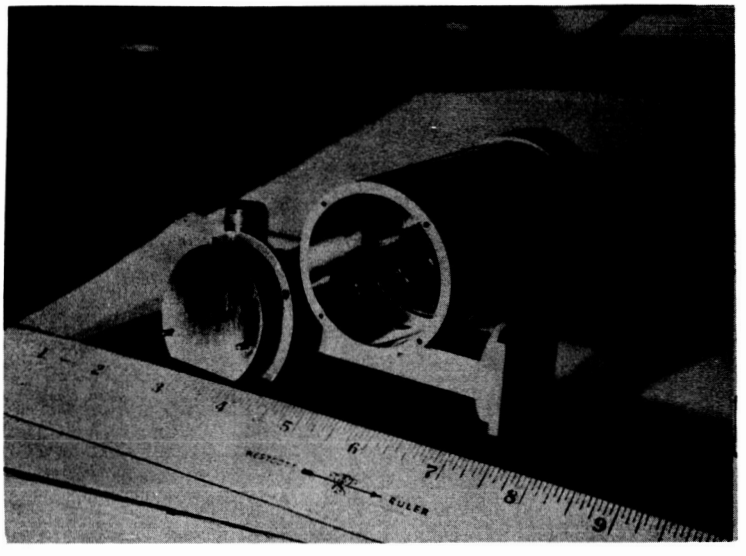


FIGURE 64

OPTIMUM RESPONSE CAVITY

of the sum of the reciprocals of the Q's of the individual system components calculated to include the effects of loading. Since the Q of the cavity itself appears in the formula as a reciprocal, the extremely high Q of commercial cavities (of the order of 10,000 to 100,000) could be reduced considerably before much effect on the overall Q would result. It is felt that a cavity-horn combination made from metal-coated molded plastic might have a sufficiently high Q for the present application. No model of such has been constructed. This would be a fruitful field for further investigation.

Since some of the cavities might be relatively inaccessible, data were taken using different lengths of coaxial cable feeding a remote cavity. Using RG-58A/U coax, lengths up to 20 feet were used without too much detrimental effect on the response. Beyond this length of coax, the ringing time was such that it became difficult to distinguish response and noise. The results of this experiment are displayed in Table V.

TABLE V

Coaxial Cable Length	Pulse Width
0 feet	3.2 microseconds
6 "	3.4 "
12 "	3.0 "
18 "	2.8 "
24 "	2.3 "
30 "	1.8* "

*Unstable.

Note that in Fig. 60, with the cavity tuned to a frequency of 9,240 MC, and in Fig. 62, with the cavity tuned to a frequency of 9,247 MC, the response is practically nil. The estimated bandwidth between half-power points is of the order of 2 mc; thus with a swept frequency radar many such horn-cavities could be operated with the same source.

For missile application it is evident that the optimum cavity configuration will be that which combines a maximum response with minimum cavity dimensions. The optimum device implies maximum Q with minimum volume. Calculations using formulas and charts in the reference show this optimum design for a right cylindrical resonant cavity in the TE_{011} mode at 9,245 MC to be: L = D = 4.27 cm. This cavity is shown in Fig. 65. The material chosen was aluminum because of its light weight and reasonably good conductivity. This cavity, with a simple screw tuning adjustment for frequency separation, could be used throughout.

Tunnel Diode Triggered Oscillator - the Active Approach

The active approach to the information problem in which power sources of small size and low cost, such as penlight or hearing aid cells, are included in the transducer assembly, has much to recommend it. The interrogating radar with its relatively high energy pulses is obviated. Only the sensitive radar receiver is needed.

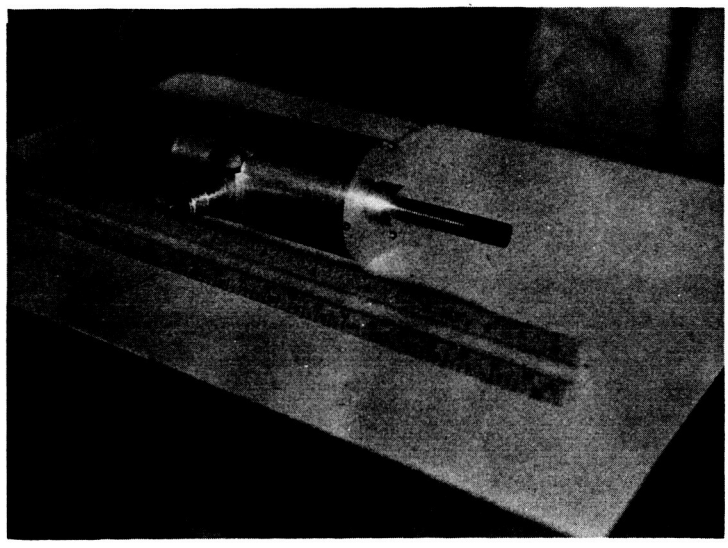


FIGURE 65

ALUMINUM CAVITY

The specifications impose some rather severe restrictions on the usable devices; as a consequence of the requirement of low cost and low weight, energy drain must be minute. The system must also generate sufficient output power, be sharply tunable, and oscillate at high enough frequency for high directivity of the receiver system. These requirements indicate quite clearly a device such as a low power-consuming microwave tunnel diode.

While the final model should probably be designed to operate in the X- or K-band to satisfy the size and weight requirement, due to the much higher cost of tunnel diodes operating in this region, the system set up for study was designed to oscillate at 1,000 mc. This system is illustrated in Fig. 66 and Fig. 67. It consists essentially of a 1,000 mc., IN2940 tunnel diode, D_1, a back-biasing silicon diode, D_2, a biasing resistor, R_d, and a filter capacitor, C, a coaxial cavity resonator shown schematically in the dotted box, a battery, E, and a sensor, R_s, which exhibits a change of resistance under the action of the leaking tracer. A Hewlett-Packard 440-A detector is used to check oscillation of the cavity. Table VI indicates the maximum value of the sensor resistance which will just permit oscillation of the system for various values of the supply voltage, E.

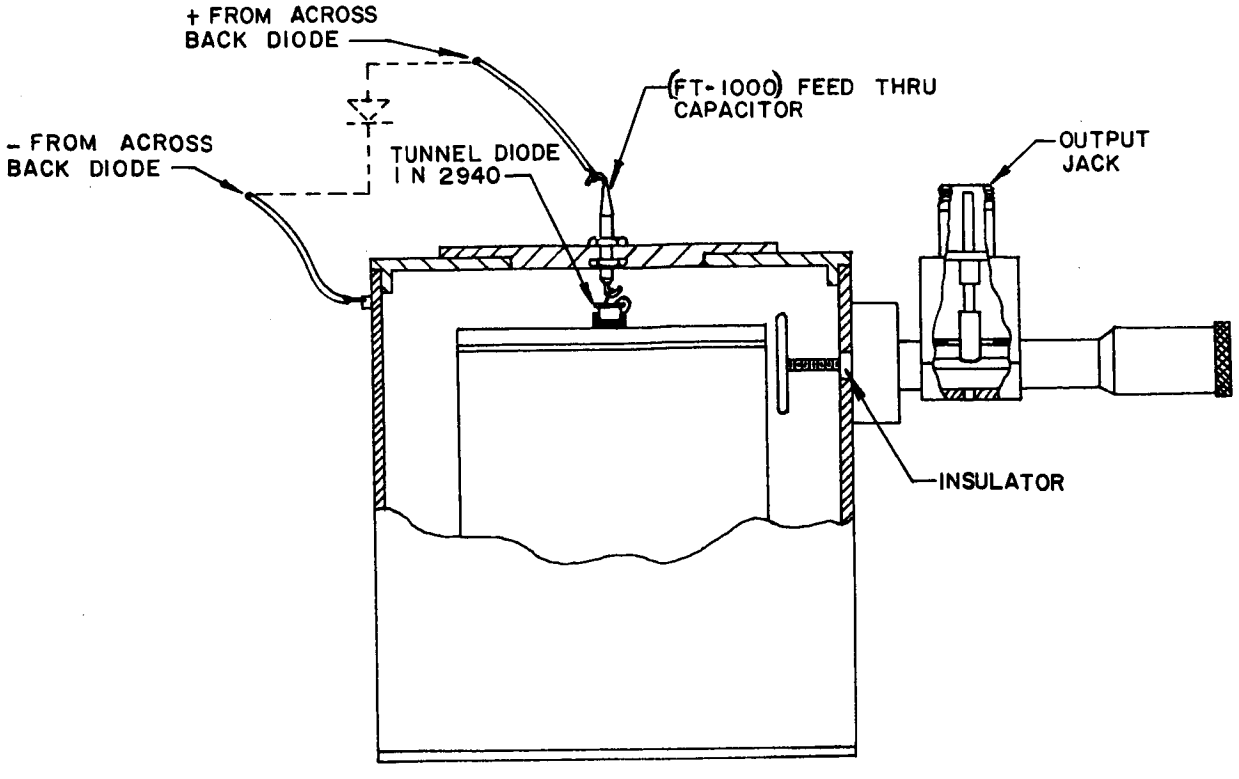


FIGURE 66

TUNNEL DIODE OSCILLATOR SCHEMATIC

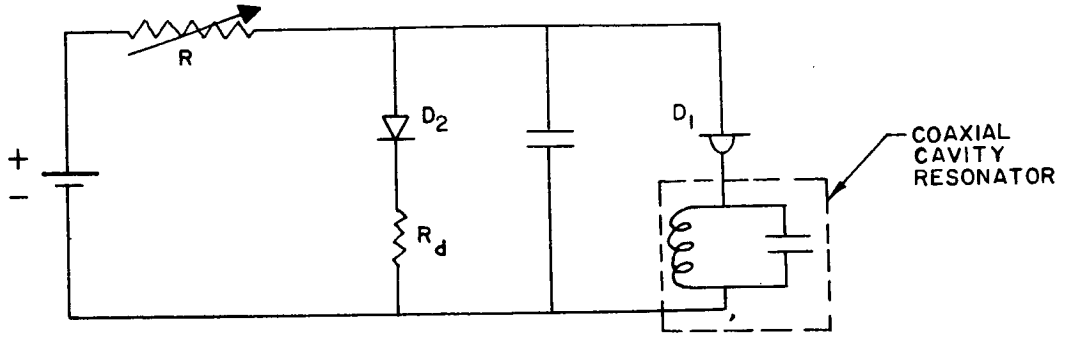


FIGURE 67

TUNNEL DIODE CAVITY OSCILLATOR

D_2	BACK BIASSING SILICON DIODE
D_1	I N 2940 TUNNEL DIODE
C	.001 MFD FEED THROUGH CAPACITOR
R_d	68 Ω RESISTOR

TABLE VI

CRITICAL SENSOR RESISTANCES FOR TUNNEL DIODE OSCILLATOR AT VARIOUS SUPPLY VOLTAGES

Supply Voltage	Detector Resistance
1.5 volts	1100 ohms
3.0 "	2500 "
6.0 "	5200 "
8.0 "	7000 "
10.0 "	8600 "
12.0 "	10000 "
14.0 "	12000 "
16.0 "	14000 "
18.0 "	15700 "
20.0 "	18500 "
25.0 "	22000 "
30.0 "	27000 "

Transducers

The transducer sensitive element must control the oscillation of the frequency-determining cavity in either the passive or the active system. One of the most promising switching devices is the solid-state detector actuated by alpha or beta particles originating from radioactive tracers. Krypton, polonium and radon have been considered as possible radioactive tracers. Both polonium and radon are good alpha particle

sources but unfortunately are undesirable from the health physics standpoint. Due to this difficulty we were advised not to pursue this area any further.

The chemical reactant tracer method also appeared quite promising, and a newly discovered compound designated as TCNQ (described in Chapter IX of this report) was given considerable testing. One approach, which time and money on the present contract did not permit, would be to construct a membrane coated with TCNQ and build it into the cavity of the passive system described above. A nearby leak of gases containing tracers of triethylamine or quinoline would cause a chemical reaction with the TCNQ and change the membrane from an insulator to a conductor. This could be used as a switch and either cause the system to oscillate or to cease oscillating as described.

Other methods which might be explored include ionized gaps, ferrites, and variable conductivity tapes and ceramics.

Some idea of the values of elements needed if the TCNQ sensor is to be used with the active system may be had from the following tunnel diode triggered oscillator study.

Fig. 68 shows plots of the characteristics of the 1N2940 tunnel diode, the biasing diode, the parallel equivalent of the 1N2940 and the biasing diode, and the parallel equivalent of the 1N2940 and the resultant of the biasing diode in series with a 68 ohm resistor, R_d.

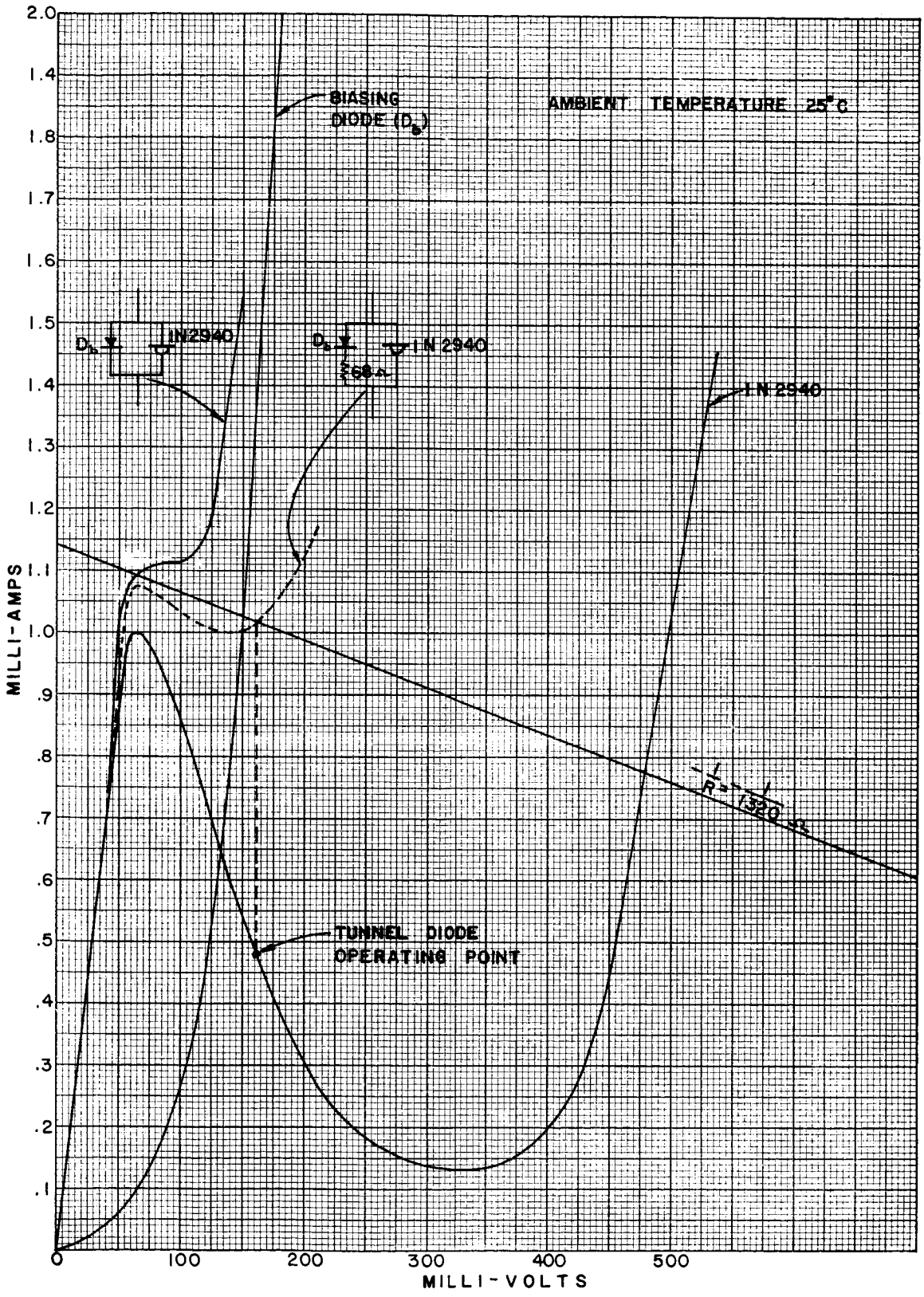


FIGURE 68

TUNNEL DIODE COMPOSITE
CHARACTERISTIC PLOTS

The dotted curve shows the best resultant for switching into the negative resistance region of the tunnel diode.

Fig. 68 shows that there is no output with E = 1.5 volts when R_s = 1340 ohms. When R_s is reduced to 1320 ohms the system oscillates since the tunnel diode is biased in its negative resistance region (V_d = 160 mv, I_d = 0.48 ma).

As R is further reduced, the amplitude of oscillation gradually increases. If the 68 ohm resistor, R_d, is omitted, the resistance, R_s, (representing the TCNQ grid) would have to be reduced to 1280 ohms to cause the system to oscillate weakly.

CHAPTER IX

MISCELLANEOUS DEVICES

Certain devices and ideas encountered in the course of this study have appeared to have considerable promise but have not been sufficiently developed to yield usable results. These ideas are reported here in the hope that they may some day yield to more intensive investigations than this project has been able to afford them.

The Shrouded Halogen Detector

An effort was made to develop a practical halogen detector which would avoid background contamination by flushing the probe with clean air. A variety of configurations and air flows were tried (Fig. 69). While this technique did succeed in reducing the background signal appreciably, the signal remained unpredictably variable in strength making detection of leaks in background a practical impossibility.

It was also noted that the purge air flow made necessary placing the probe tip directly against the source of leak, which is sometimes impossible in confined-space.

The considerable range of shroud geometry and air flow rates tested, and the mediocre results, lead to the conclusion that the shrouded halogen detector with purge air leaves little promise for the future.

The original plan which called for a small flush tank over the

nozzle of the General Electric Halogen Detector, Model H-2, was modified when it was found that the cooling ports located on top and bottom of the detector gun were also sensitive to halogen contamination. When high contamination was present the indicator was deflected full scale even when the nose of the detector was in a halogen free atmosphere. This was traced to the cooling port sensitivity. It was, therefore, necessary to construct a flush chamber that would keep contaminated air away from both the nozzle and the cooling ports.

A clear lucite flush tank, similar to the front part of the tank shown in Fig. 69 had already been constructed to study the air flow and flush characteristics of the flush system. The first attempt to flush the cooling ports was made by placing a polyethylene bag over the entire gun so that only the nose was free. The uncontaminated air was then circulated through this bag to cool the gun. Due to the high temperatures of the surface of the gun which caused the bag to melt this method was discarded.

A three inch outside diameter lucite tube blocked at one end with a lucite plate was slipped over the front part of the gun. The small flush tank was glued to the front face of the lucite plate as shown in Fig. 69. A single hole was drilled in the bottom of the large lucite tube to supply air for both flushing the nose and cooling the gun. This did not allow for variations in the flow used in the

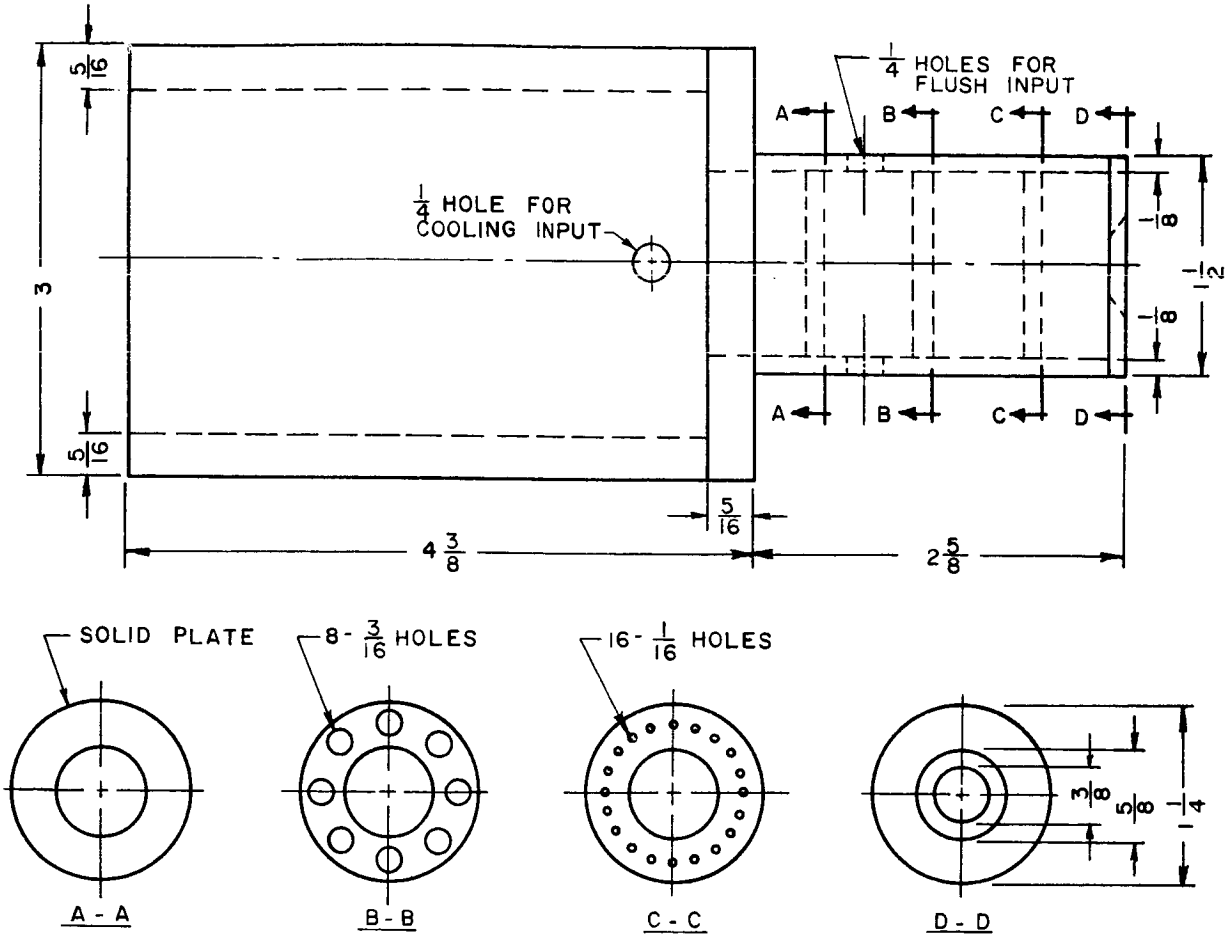


FIG 69

HALOGEN DETECTOR FLUSH TANK

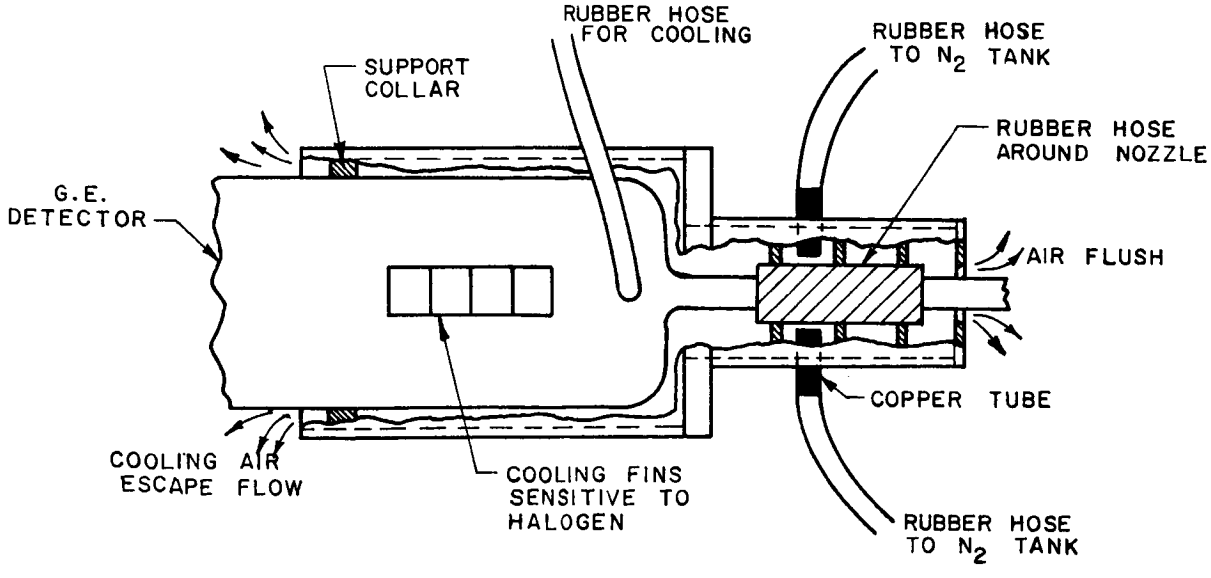


FIG 70

FLUSH TANK AND G.E. DETECTOR

flushing process so the solid plate (1) was added as were two holes in either side of the smaller chamber to facilitate variation of the flush air flow.

The front of the flush tank was supported by the nozzle of the detector gun which was passed through a snug fitting piece of rubber hose as shown in Fig. 69. Because of the slight taper of the nozzle this gave an airtight seal when the nozzle was pushed firmly in place. The back of the tank was supported by a loose fitting collar (see Fig. 69) which was attached to the gun but which was machined so that it would slide inside the large lucite tube. This allowed easy positioning of the nose of detector with respect to the front face of the flush tank.

Plates (2) and (3) (cf. Fig. 69) of the small lucite tank were constructed to equalize the air flush around the nozzle in such a way that no cross currents were obtained. To aid further in the solution of this problem two input tubes were used for the flush tank, one on either side of the lucite tube.

Tests made with various sized and shaped holes in the end face plate of the flush tank (plate (4) in Fig. 69) showed that the best design was one which tapered as shown in the figure. This gave a good uniform flow of flush air around the nozzle of the detector.

Fig. 70 shows the shroud in position on the detector.

The Hot Ceramic Detector

In 1954, W. C. White[2] of General Electric Research Laboratory in Schenectady, N.Y., published a description of a leak detector for inflammable gases based on what appeared to be positive ion emission from a heated platinum wire in contact with a warm ceramic cylinder charged with alkali metal salt. According to the report this detector was capable of locating a leak of a few parts per 10,000 of H_2 or a leak rate of about 0.016 SCIM.

The advantages of such a detector lie in its small size, adaptability to existing circuitry (GE Halogen detector unit), low temperature of operation and sensitivity to low molecular weight gases such as H_2, CH_4 (methane), and C_2H_6 (ethane) which will not pool but diffuse rapidly throughout the system.

Its disadvantages lie in its low sensitivity, lack of reproducibility of results (making it qualitative rather than quantitative) and, perhaps, the inflammability of the gases to which it is sensitive. The leak rate mentioned above appears to be the leak which could be detected with 100% tracer and while greater sensitivity is claimed for the higher molecular weight gases in the series, this is still too rich a mixture to be taken seriously in the present application.

Since no further mention of the device has been found in the literature searched, and since the General Electric Company does not

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[2]Superscript numerals refer to references at end of this chapter.

offer such a device for use with its Halogen detector, it is presumed that the ceramic detector can not be considered in its present form to be competitive with the Halogen detector.

Since no satisfactory detailed explanation of the principle of action of the device was found in the literature, it was decided that a theoretical investigation of the behavior of hot wire-warm ceramic conduction cells should be undertaken.

A mixture of aluminum silicates known to produce good uniform easily moldable ceramics at reasonable temperatures was obtained and some samples made up. Early qualitative tests on these samples with small leaks through capillary tubes showed them to be somewhat sensitive to H_2 and CH_4 (methane). However, the samples thus crudely checked seemed to be more sensitive to Argon and Helium, than to the inflammable gases.

Since these first exploratory qualitative tests suggested that other than inflammable gases might be used and since so little was known about the principle of operation (or principles since such dissimilar agents as H_2 and He might easily be acting in very different manners to produce the same results) a series of experiments was undertaken to try to accumulate sufficient reproducible data to permit a theoretical study of the phenomena involved.

The variables selected for first study were: a) the impregnants, b) the hot wire temperature (and its concomitant, the warm ceramic

temperature), c) the nature of the tracer gas, d) the concentration of the tracer gas, and e) the temperature at which the ceramic was fired.

Impregnants used included NaCl, both as commercially available table salt (Morton's) and chemically pure reagent, Kl, NaI, NaBr, CsCl, LiCl, KBr, and dextrose. Of the above, commercial table salt proved the most effective impregnant. Its listed constituents, NaCl, sodium-silico-aluminate, dextrose and KI, taken separately and in various combinations have not approached the performance of the table salt as available on the market. As pointed out by White[2] reproducible data in this field are hard to obtain. Table VII summarizes qualitative results so far recorded.

TABLE VII

Impregnant	Sensitivity	Reproducibility
NaCl pure	low	poor
NaCl commercial	good	fair
KI	low	poor
NaI	medium	very poor
NaBr	low	poor
CsCl	low	very good
LiCl	low	fair
KBr	low	poor
Dextrose	low	poor

Hot wire temperatures, calculated from the volt-ampere characteristic of the platinum wire and its temperature coefficient of resistivity range from 300° C upward to around 1000° C. Current was found to be a monotonic function of hot wire temperature over the range investigated.

Two tracer gases were readily available for study. These were argon and helium, both noble gases. In order to remove uncertainties due to varying moisture content and possible other contaminants in the laboratory air, pure dry nitrogen was used for admixture with the tracers. Known composition mixtures were obtained by admitting tracer gas to an evacuated space (under a bell jar on a vacuum plate) until the pressure reached the partial pressure of the gas in the required mixture and then diluting with pure dry nitrogen to atmospheric pressure. By subsequent reduction of the pressure by means of the vacuum pump and redilution with nitrogen, any required mixture could be obtained. Fig. 71 is representative of the results obtained with argon. Fig. 72 shows similar results for helium.

Results presented in Fig. 71 and Fig. 72 also show the effect of varying the firing temperature in producing the ceramic portion of the detector. Firing temperatures used were 1450° F, 1940° F, and 2040° F. Other specimens were fired at higher heats but proved insensitive because of a surface glaze which develops at these heats.

10% Table Salt Specimen, Cylindrical

Baking Temp.: 2040°F-1940°F-1450°F
Operating Temp.: 800°C
Bias Voltage: 330V

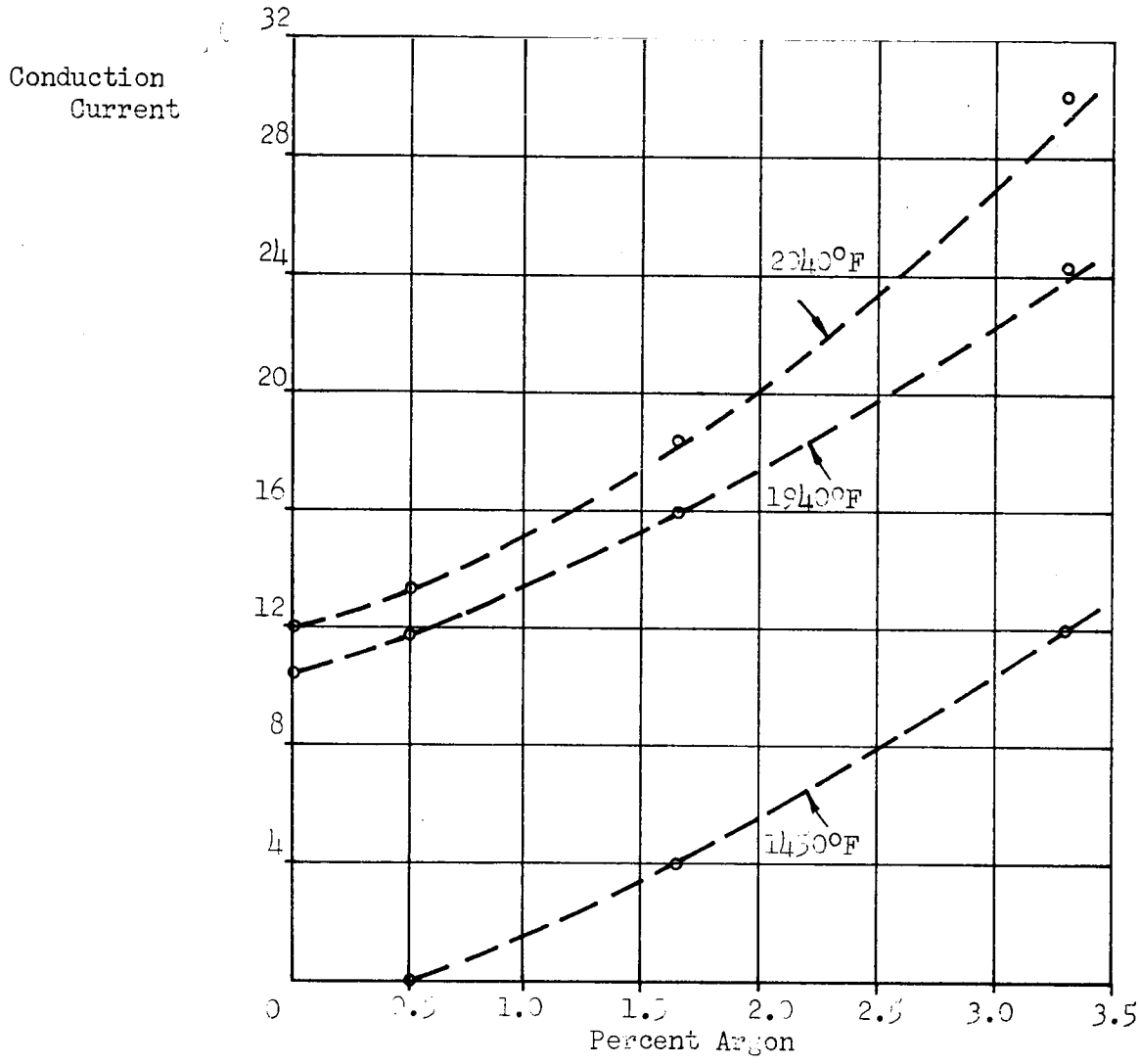


FIGURE 71

HOT CERAMIC DIODE RESPONSE-ARGON

Cylindrical Table Salt Specimen

10% Table Salt
Operating Temp.: 800°C
Baking Temp.: 2040°F-1940°F-1450°F
Bias Voltage 33V

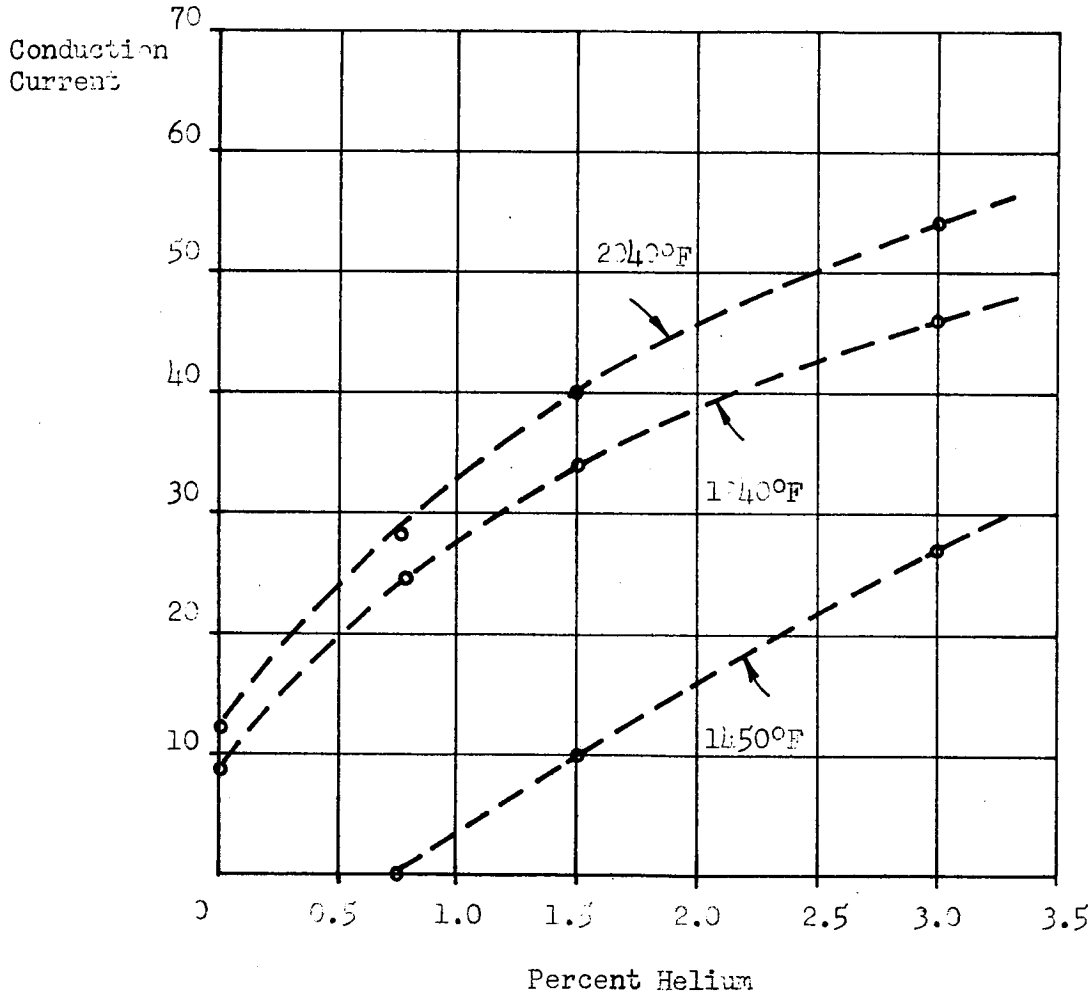


FIGURE 72

HOT CERAMIC DIODE RESPONSE-HELIUM

Sanding to remove this glaze did not produce satisfactory results.

Ceramic specimens were formed in two general shapes. One of these correspond roughly to the simple cylinder used by White [1]; the other was in the form of a hollow small angle cone. Contact with the body of the ceramic was by means of a short piece of platinum wire embedded in the material. The hot wire was wound around the outside of the cylinder, but was wound into the inside of the cone. The purpose of the inverted structure of the conical design was to assure good contact between the expanded hot wire and the surface of the cone of ceramic. These shapes are indicated in Fig. 73.

The very simple circuitry used in the tests is indicated in Fig. 74.

Nothing approaching the sensitivity of the halogen detector has been noted. Fig. 75 and Fig. 76 show the test setup used.

Mercaptan Sniffing

In view of the difficulty foreseen in injecting sufficient amounts of tracer gases into high-pressure control-gas containers, the research group believes that the mercaptans should be seriously considered for leak detection. Due to the extreme sensitivity of the human nose to mercaptan, also its safety in small concentrations and to its lack of corrosiveness when used in small concentrations and for reasonably short periods of time, and finally to the extreme simplicity of the detector (human nose), this might indeed be one very workable solution to the leak detection problem.

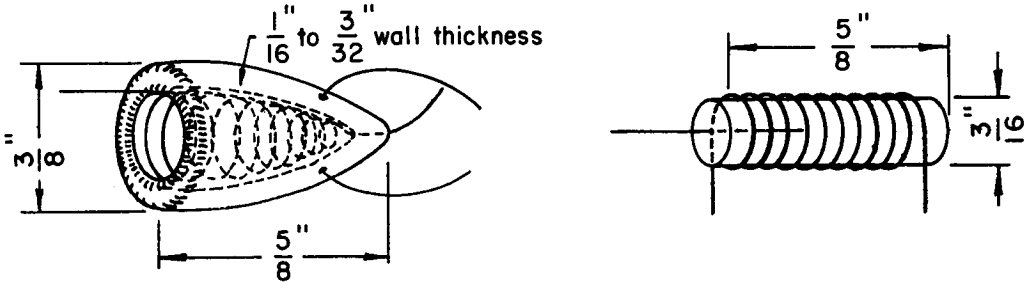
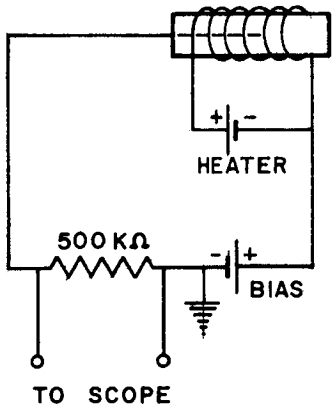


FIGURE 73

CERAMIC DETECTOR GEOMETRY



Heater: 0-5 volts 0-2.5 amps

Bias Voltage: 250 - 380

FIGURE 74

CERAMIC DETECTOR CIRCUIT

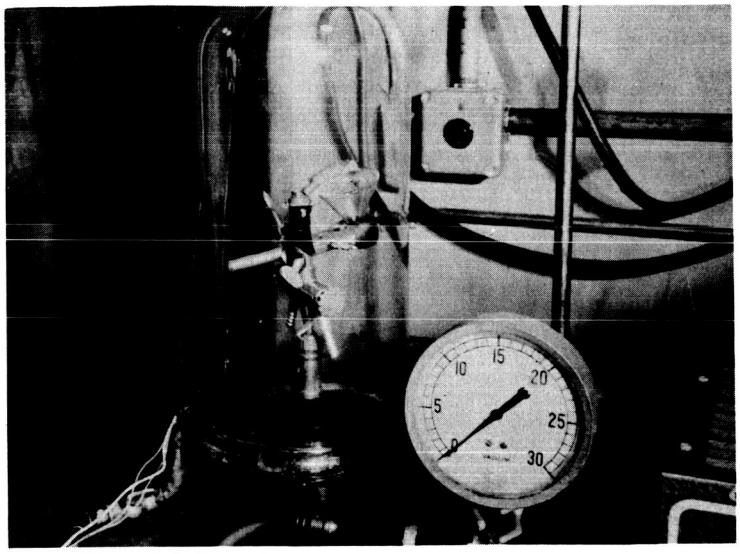


FIGURE 75

TEST APPARATUS FOR CONTROLLED MIXTURES OF ARGON-NITROGEN AND HELIUM-NITROGEN

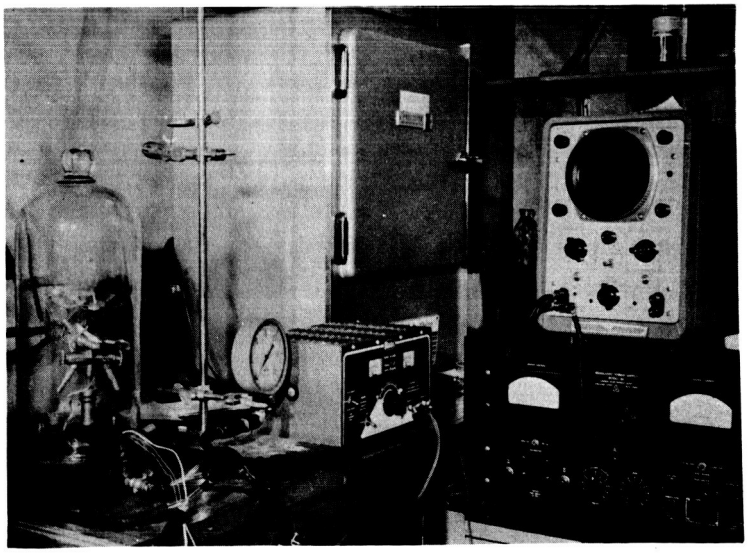


FIGURE 76

TEST SET-UP FOR STUDY OF CERAMIC DETECTORS

Since little is known about the reliability of the human nose as a detector onboard a flight vehicle, it must be pointed out here that further study would be necessary if this method is to be considered in such applications. This phase of space medicine is beyond the competence of this group.

Ethanethiol (ethyl mercaptan) has been extensively used as an odorant in natural gas systems for the purpose of leak detection[3]. Munyan reported[4] positive leakage tests with concentrations of 0.81 parts of ethanethiol per million of natural gas. Ethanethiol has also been utilized in refrigerant systems for the same purpose[6]. These uses are possible because of the very low concentrations which are nasally detectable. Thomas reports[7] that ethyl mercaptan is "detectable" at 0.002 parts per million in air, "distinct" at 0.03 - 0.07 ppm, and "very strong" at 0.6 ppm.

Methanethiol (methyl mercaptan) has not been used for such purposes or at least no references to such have been found. On the contrary, the compound is noted to be an odor problem arising from sulfite treatment of wood pulp and the emphasis here has been on eliminating the offensive odor of the material in such areas. Reid notes that the degree of unpleasantness of mercaptan odors decreases with increasing molecular weight; thus methanethiol is the most obnoxious of the series.

Methanethiol is detectable in very small quantities. Freudenberg and Reichert[8] note that one part of methanethiol in 4.5×10^9 parts of air (by volume) is a maximum level beyond which the odor becomes easily detectable and unpleasant. Comparing to the data reported above for ethanethiol, this value corresponds to a concentration of the mercaptan of 0.0002 ppm.

Properties of Mercaptans

A. Toxicity. So-called "threshold limit" values have been recorded[9] for various gases and vapors. Ethanethiol appears on a tentative listing which suggests that a concentration of 250 ppm by volume in air (or 640 mg per cibic meter) is the maximum safe level for day-by-day exposure without harmful effects. On the same listing methanethiol is given the values 50 ppm and 100 mg. per cubic meter. Thus the latter is somewhat more toxic than ethanethiol. These values far exceed the required concentrations for nasal detection and suggest no toxicity problem. Fongauz, in discussing hygiene problems associated with recovery and treatment of high-sulfur petroleum (which contains significant quantities of mercaptans) notes that mercaptans do not pose a toxicity problem under these circumstances[10].

B. Physical Properties. The most complete reference for physical properties (as well as chemical properties and other areas of interest) of mercaptans is Chapters I and II of Volume I of the series by

E. E. Reid, Organic Chemistry of Bivalent Sulfur.[3] Many properties are reported here and references for others are given. Haines has reported data on ethyl mercaptan for highly purified material.[11] A number of properties are reported here from direct measurements and others as derived from these measurements.

A number of authors provide measured and calculated data on methanethiol.[12, 13, 14] Oldach and Field provide values[15] for the vapor pressures of both methanethiol and ethanethiol in ten degree increments from -20° to 40° C. For example, they report at 20° C. values of 1.68 and 0.502 atm. for methanethiol and ethanethiol, respectively.

Such high values suggest that even at so high a total pressure as 2000 psi either of these two materials would be present at a significantly higher concentration than the minimums required to allow nasal detection. Conversation with Dr. Wendell V. F. Brooks of the Department of Chemistry, Ohio University, suggests, however, that some problem may exist at such high total pressures with a slow diffusion rate, particularly through narrow diameter piping. If, for example, a sample of mercaptan were placed in a closed system at atmospheric pressure and then the system pressurized to 2000 psi, the mercaptan sample might be trapped in one end of the system and momentarily have its partial pressure in the localized area exceed its vapor pressure and

thus partially liquefy. Diffusion away from this localized area would be required in order adequately to fill the system with odorant.

C. Chemical Properties. Mercaptans can be oxidized and sulfur appears as sulfur dioxide in many such reactions. This product is highly corrosive and presents an obvious problem. In addition, the possibility of oxidation leads to an explosion and flammability problem, potentially.

Jones reports the following data,[16,17] the limits of inflammability of ethanethiol are 2.80 - 18.2% by volume in dry air. All mixtures within these limits are inflammable at laboratory temperature and pressure. Ignition temperature is 299° F. in air and 261° F. in oxygen. The flash point is -40° F. He observes that the hazards are multiplied in compressed air lines and suggests the use of 5 parts of Freon 12 to one part of ethanethiol or 5.3 parts of Freon 22 to one part of the thiol (by weight) in order to provide non-inflammable odorant mixtures at 70° F. and one atmosphere pressure. For higher temperature and pressure conditions (such as 90 psi and 200° F.) he suggests a 10% increase in the Freon proportions.

Cullis and Roselaar[18] report that the slow combustion of methanethiol and ethanethiol in air begins in the temperature range 200 - 250° C. The rate of reaction is accelerated by increasing the oxygen concentration. Thus it would appear that a room temperature operation with low concentrations of mercaptan, in nitrogen or other

inert gas or even in air itself should not pose any great oxidation problem. Surely pure oxygen systems would very likely constitute a hazard.

Corrosion by mercaptans. The reports relative to this question are somewhat contradictory. Henderson reports[19] no effect on copper by ethanethiol in naphtha solutions in the "distillation-corrosion" test, in which copper strips are exposed to refluxing mercaptan and solvent at 94° F. On the other hand, Wood reports[20] that copper and brass were corroded by mercaptan solutions in naphtha. McKay and Worthington[21] report (page 426) that gasolines at 80° F. having sulfur compounds up to 0.01% sulfur do not tarnish copper. Gasolines having 0.02 - 0.06% sulfur do show increased tarnishing but little penetrating corrosion. They also note (page 242) that sulfur compounds are an important variable in corrosion of iron and steel upon exposure to atmospheric conditions. Reid[3] refers in Chapter 2 to the copper strip test for detecting mercaptans in petroleum and notes that control of time and temperature is essential to proper results from varying test conditions.

Eigenson and Toporova found[22] no evidence of corrosion of copper by ethanethiol in 0.003 - 2.4% solutions in benzene and n-heptane. Ginden reported[23] no effect on iron by mercaptan solutions in cyclohexane.

On the other hand, U.S. Patent 2,807,592 (noted in Chemical Abstracts, 52, 701g) suggests the use of 0.003% by weight of mixtures of guaiacol and secondary amines with mercaptan odorants to inhibit corrosion and oxidation.

Considering the variance of these reports and the lack of information on systems comparable to the one under consideration here, a specific effort to measure the corrosive effect of these mercaptans on the metals with which the odorant would come in contact and at the concentration, temperature, and pressure of the prospective systems, seems required.

D. Availability. Methanethiol and ethanethiol are available in laboratory quantities from Eastman Kodak and in larger quantities from the following suppliers: Methanethiol: Amoco Chemicals Corporation, Wallach-Gracer Export Corporation, and Pennsalt Chemical Corporation; Ethanethiol: Mallinckrodt Chemical Works, McKesson and Robbins Chemical Department, Phillips Petroleum Company, Oronite Division, Pennsalt Chemicals Corporation.[24]

Methods Involving Radioactivity

Radon-222 also called Emanation-222 or radium Aα(Rn-222 or Em-222) has a half life of 3.8 days (α decay of 5.5 Mev) and is produced by the decay of 1620 years radium-225. Radon is a noble gas, completely inactive chemically.

The plan for leak detection would be to mix the radon with nitrogen and then pressurize the system being checked with the nitrogen and radon mixture. Leakage of the radon would be looked for by solid-state α particle detectors. The output of each detector is to be transmitted by the microwave system to a central clearing house and operator.

Work on the microwave system and solid state detectors is described elsewhere in this report. The solid state detectors are available commercially, and can be made sensitive to α particles only, and almost completely insensitive to either B particles or γ rays. Background counting rates of 1 to 5 counts per minute can be expected.

A calculation of the amount of radioactivity needed is based on the following considerations:

(a) The area of the solid state detector sensitive to α particles is assumed to be 0.33 square centimeters.

(b) Range of 5.5 Mev α particles in air is 3.5 centimeters.

(c) Pulses greater than due to the last 0.5 centimeter range of the α particles are to be counted.

(d) The gas at the surface of the detector is 1% of the original radon mixture. (This corresponds to the approximate dilution through diffusion occurring at 10 inches from a 1 SCIM leak for radon.)

(e) A counting rate of 1 count per second is to be recorded by the detector (10 to 60 times background).

Under these conditions the ratio of the number of radon atoms to nitrogen molecules would need to be 4×10^{-12}. This corresponds to 6 millimicrocuries per cubic centimeter or 170 millicuries per 1000 cubic feet of nitrogen (0° C, 1 atmos).

One curie of radon is generated (in equilibrium) by one gram of radium. The suggested method of charging the nitrogen with radon is by a slug of radium kept in the nitrogen storage tanks. One hundred seventy milligrams of radium then will keep 1000 cubic feet of nitrogen charged to the above concentration, the system coming to equilibrium in 3 or 4 radon half lives or about 14 days.

The choice of one count per second above as a required counting rate was more or less arbitrary. The background counting rate is expected to be less than 0.1 counts per second and should not be the deciding factor. Rather the criterion is the operator's ability to notice an increased counting rate and one count per second could not pass unnoticed.

The most serious disadvantage of the use of Radon-222 is the residual radioactivity that will be left in the system after the radon is removed. Each Radon-222 that decays in the system will leave behind an atom of lead-210 (radium D) with a half life of 22 years having α, β, and γ activities. How much activity is left behind will depend on how long the radon is in the system; but in general, if the radon is in long compared to its half life (3.8 days) then each millicurie of radon

will leave behind 2 microcuries of lead-210. (Decontamination of the lead-210 can be achieved by scrubbing with an organic free radical.)

Polonium-210 is available from the decay of radium-226 or can be produced by neutron absorption on bismuth-209. Polonium-210 gives off a 5.3 Mev α particle with a half life of 138 days.

Since polonium-210 is an alpha emitter like radon-222, all the calculations for radon apply to polonium-210 (i.e., 170 millicuries of polonium-210 per 1000 cubic feet of nitrogen would give a counting rate of one count per second on the same detector). The advantage of polonium-210 over radon-222 is that there will be no residual activity following the polonium-210 decay (lead-206, the daughter, is stable). The chief disadvantage of polonium-210 is that it is not a gas nor is it known whether or not it can be combined chemically to form a gas (or vapor), although certain polonium organic compounds are known to be highly volatile, especially diphenylthiocarbaside.[25]

Health Considerations

(a) Radon-222. The maximum permissible concentration (m.p.c.) for radon-222 in air breathed by personnel is 10^{-8} microcuries per cubic centimeter[26] or radon-222/air = 6.6×10^{-18} by volume. In terms of the maximum permissible concentrations, then the proposed test gas would be charged with radon to about 100,000 m.p.c.

(b) Polonium-210. The m.p.c. for polonium-210 is 2×10^{-10}

microcuries per cubic centimeter,[26] or polonium-210/air = 4.4×10^{-18} by volume. In terms of the maximum permissible concentration, the proposed test gas would be charged with polonium-210 to about 30,000,000 m.p.c.

It is clear from the above that these isotopes could only be considered for leak detection by remote operation. There may be some systems that can be leak checked with radon during an actual firing, either out on the test stand or at launching, but certainly these isotopes are not practical for routine testing in a confined region. This method may also be useful for leak testing in space, again when no personnel are involved.

Vibrating Capacitor Electrometer

When an air capacitor has plates with different surfaces, one treated to stabilize it against adsorption of gas, and the other either cleaned metal or metal treated to encourage preferential adsorption of certain polar molecules, a potential develops across the plates. If the plate spacing is caused to change rapidly and periodically, an A-C voltage is generated which can be amplified and then synchronously rectified and measured. If this voltage is nulled out by means of an adjustable D-C supply, for a given air composition and flow rate between the plates. the change of composition and/or rate of flow can be very sensitively detected as an output voltage. Non-polar molecules (e.g., benzine) have orders of magnitude less than polar molecules.

This system seems to have the following advantages over commonly available detectors:

1. It should be possible to seed the system under test with some polar trace gas or vapor and no radioactivity need be used.

2. By proper treatment of the plate, various tracers can be made to give differentiable responses. This suggests that a dual sensor could be designed to give some idea of the location of a remote leak.

3. No high temperatures or voltages are required in the sensing element; therefore, no explosion or fire hazard.

4. This is a relatively simple system which would encourage miniaturization through the use of solid state devices.

5. In reported results, this device can detect concentrations of one part in 10^7 of polar molecules in clean, dry air.

In view of the foregoing, the staff feels that this device deserves considerable more study.

TCNQ Detectors

In September 1962, our Chemistry research group read several preliminary reports from DuPont concerning the preparation of a complex anion-radical salt which has an unusually high electrical conductivity. This salt was formed by bringing a compound called tetracyanoquion-dimethane (TCNQ) into contact with a reactant, triethylamine (TEA). The resulting anion-radical salt, $Et_3NH^+(TCNQ)^-_2$, ranging in yield

from 50 to 90% of one externally supplied proton and electron per molecule as its formula illustrates. The high conductivity property of the salt aroused interest regarding its applicability as a leak detector.

Sufficient quantities of this compound were synthesized by our group, and a number of postage-stamp-sized interlacing grids of copper or aluminum were constructed and covered with a thin layer of TCNQ. The reactant, TEA, was brought into contact with the TCNQ layer by bubbling air through liquid TEA. This mixture of air and TEA was then passed over the grid for a variable period of time, and changes of resistance were measured.

After considerable practice and improvement in the art of manufacturing grids, it was found that using a 6% mixture of TEA with air, an irreversible reaction took place which converted the thin layer of TCNQ into a multi-path conductor between the teeth of the interlacing grids. A very important effect noted was the integrating property of the reactant which permitted the detection of a small quantity of TEA if the leak persisted long enough. In other words, repeated exposures increased the grid conductivity.

For a time it was thought, by the DuPont group and by our own research team, that an impurity in the TEA might be the main agent of the chemical reaction. This possibility gave rise to the hope that a very minute quantity of TEA impurity would be sufficient to cause a

change in the conductivity of the TCNQ. Later study in the case of quinoline indicated that the impurity acted more as a catalyst which furnished the extra electron and proton needed in the reaction. In the case of triethylamine, while excess TEA molecules can serve as sources for the necessary protons and electrons needed in the reaction, the impurity in the amine acts as a better provider than the TEA itself. In any case, it is quite clear from the yields mentioned earlier that the amine itself is the actual reactant.

The difficulties encountered were mechanical and electrical rather than chemical. Many attempts were required before a reasonably reproducible TCNQ sensor could be fabricated that also was relatively insensitive to water vapor. Unfortunately the bakelite backing used originally to support the TCNQ and the metallic grids was found to have an affinity for water vapor. Thus the water present in the TEA (Et_3N) lowered the resistance of the sensor and masked-out the actual change of resistance due to the chemical reactant in our earlier attempts. It was this moisture effect that led the group to believe at one point that the reaction was reversible.

It should be pointed out that TEA forms a azeotrope with water which makes their separation very difficult. Hence, all TEA normally will have this water content unless given special treatment.

The other major difficulty encountered in fabrication was to

develop a sputtering process so that an aluminum grid could be formed over the TCNQ layer to allow continuity between the grids and their wire contacts. Glass backing was found to be superior to bakelite from the water vapor standpoint.

The major obstacle still existing to the application of the TCNQ method to missile leak detection remains the relatively large concentration of TEA needed to produce useable resistance changes in the detector within reasonable time limits. The concentrations required in the test gases preclude the use of TEA-TCNQ in high pressure applications at least so far as present results indicate.

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CHAPTER X

BIBLIOGRAPHY

Literature Search

Literature published within the past thirty years has been carefully scanned for articles and papers dealing with leak detection techniques and related topics. From the Chemical Abstracts, Science Abstracts, Nuclear Science Abstracts, and Electrical Engineering Abstracts, papers published prior to January, 1961 were located. More recent papers were found by surveying the contents of approximately 100 technical and scientific journals. In addition, many reports have been obtained from ASTIA (Armed Services Technical Information Agency). As a result of this survey approximately 1000 papers and articles have been abstracted and catalogued in a card file. This original card file has been color-coded according to the six separate fields of interest listed in the next paragraph.

The complete bibliography compiled to date and listed alphabetically by author follows. In the bibliography, marginal superscripts from (a) through (f) have been affixed to items of particular interest according to the code below:

(1) Mass Spectrometry (Marginal Superscript a)

Various types of mass spectrometers with design and construction details, operating procedures, and characteristics as applied to leak

detection.

(2) Infra-red and Ultra-violet (Marginal Superscript b)

Devices dependent on infra-red and ultra-violet optical properties of gases.

(3) Mechanical (Diaphragm Type) (Marginal Superscript c)

Diaphragm type pressure gauges and methods for measuring the position of a diaphragm.

(4) Other (Marginal Superscript d)

Halogen detectors, oxygen detectors, toxic gas detectors, catalytic action detectors, acoustic detectors, etc.

(5) Radioactivity (Marginal Superscript e)

Radioactive tracer techniques applied to leak detection.

(6) Conductivity High Temperature (Ceramic) (Marginal Superscript f)

Electrical and thermal conductivity type detectors, ceramic devices, thermistor bridges, and Pirani gauges applicable as leak detectors.

Bibliography entries which might have direct application to the leak detection problem were abstracted and noted by a special marginal superscript +. These abstracts are contained in CHAPTER XI.

KEY TO SYMBOLS

* = Important Publications on

a = Mass Spectrometry

b = Infra-Red and Ultra-Violet

c = Mechanical (Diaphragm Type)

d = Other

e = Radioactivity

f = Conductivity; High Temperature (Ceramic)

\+ = Article Abstracted

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AD 157534, "Evaluation of the Penn-Barton, Portable, Differential-Pressure, Fluid Flow Meter for Military Pipeline Service."

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AD 206231, "Propulsion Wind Tunnel Digital Pressure System."

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AD 261128, "The Design and Testing of Twisted Bourdon Tube Potentiometer Type Pressure Transducers."

AD 262142, "Application of a Hydrocarbon Tracer Technique to Gas Phase Mass Transfer Investigations."

AD 265197, "Final Report - 'Development of an Atmosphere Monitoring System.'"

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AD 270958, "Design, Development and Manufacture of an Electro-luminescent Pressure Transducer."

AD 271001, "Leak-Indicating Paints for Rocket-Fuel Tanks and Storage Areas."

AD 271969, "Current Vacuum Technology and Practice."

CHAPTER XI

SELECTED ABSTRACTS

Apker, L. "Surface Phenomena Useful in Vacuum Technique, Ind. Eng. Chem., 40, 846-7, 1948.

Described are the uses of thermionic emission from wires, field emission from single crystals, and photoelec. emission from W to detect very low partial pressures of gases whose presence causes current variations. Although it is not as sensitive as the thermionic method, the photoelec. method is convenient because it can be used at low temps. and with very little accelerating voltage.

Arahara, T., and Y. Takrgi. "Gas Chromatography," Abura Kagaku, 6, 32, 108, 1951.

A review with 63 references.

Baker, W. R. "Leak Detector," U.S. Atomic Energy Commission Patent No. 2,591,998, Apr. 8, 1952.

This gas-leak detector of the mass spectrometer type involves an electric circuit for periodically directing ions corresponding to the indicator gas onto the ion receiver and for amplifying the resulting periodic current, thereby providing a leak detector of great sensitivity.

Barton, R. S. "The 1441A Oxygen Leak Detector," Atomic Energy Research Estab. (Gr. Brit.) GP/M-189, 1957.

The leak detector consists of a diode with O_2 as the probe gas. Entrance of the gas into the vacuum system leads to a decay of electron emission from the hot cathode of the diode which is observed on a meter. The gage circuit is described and illustrated.

Beck, A. H., and G. King. "A Sensitive Leak Detector Using Magnetron Ionization Gages," Vacuum, 4, 147, 1954.

The construction of a differential leak detector capable of indicating leaks of 10^{-7} $\ell\mu$ sec is described. The detector uses 2 magnetron gages (cf. Ochert, C.A., 47, 7834c) in a bridge circuit. The gage is modulated at 200 cts/sec and an A.C. amplifier is used as detector. The vacuum system comprises 2 hg pumps connected to the gages through an Alpert all-metal vacuum tap. A freezing trap is placed between the 2 gages which freezes out the probe gas (butane) squirted on to the leak and introduced as the first gage

by a needle valve. The difference in butane content unbalances the bridge.

Bell, R. L. "The Omegatron as a Leak Detector," J. Sci. Instrum., 7, 269-72, July, 1956.

The omegatron, a radio-frequency mass spectrometer, has been applied to the detection of very small leaks using the helium probe technique. Although intrinsically of similar sensitivity to the conventional magnetic sector mass spectrometer, its effective sensitivity can be very much increased by reducing the speed at which the leak and leak detector combination is pumped, an operation which is not feasible with the usual mass spectrometer. By this means leaks of 10^{-11} l.mb/sec are readily found. The limiting sensitivity appears to lie in the 10^{-14} l.mb/sec region. A simple omegatron construction for leak detection is described, and precautions to be taken on the use of helium mentioned.

Bennett, W. H. "Radiofrequency Mass Spectrometer," J. Appl. Phys. 21, 143, 1950.

Uses velocity selection rather than magnetic beam deflection for gas analysis. Simpler, more compact, and more rugged than magnetic beam deflection devices. Article does not discuss application to vacuum measurement and to leak detection.

Bertein, F., C. Cherrier, L. Verot, and R. Wagner. "Photo-elastic Apparatus for the Quantitative Analysis of Coloured Gases," C. R. Acad. Sci. Paris, 230, 1866-7, May 22, 1950.

An apparatus is described consisting of a glass by-pass tube mounted on the main gas conduit, an incandescent bulb at one end of the tube and a photo-electric cell at the other end, the variations of the photo-electric current during the gas flow being amplified so as to operate an alarm relay. The apparatus has been adapted for (a) the determination and automatic recording of the content of Cl_2 or NO_2 in a gaseous mixture, (b) the determination of the humidity of a gas, and (c) the signalling of a quantity of one of the above gases, exceeding a previously fixed figure, having passed through the conduit, with an immediate stopping of the gas circulation.

Beynon, J. H., and G. R. Nicholson. "A Radioactive Ionization Gauge and Its Application to the Measurement of Latent Heat of Vaporization," J. Sci. Instrum., 33, 10, 376-80, Oct., 1956.

A radioactive ionization gauge useful in the range of pressures 10^{-5} to 10 mm of mercury, and its application to the determination of the latent heat of vaporization or sublimation of organic compounds having vapour pressures in the above range up to 200° C, are described. The instrument uses a 0.5 mc sealed radium source for ionization of the vapour and an electrometer amplifier for measuring the ionization currents produced. Values for the latent heats of fourteen compounds are given.

Bierman, A. Rev. Sci. Instr., 28, 910-13, 1957.

1. Resonance mass selector. 2. A special type of tube. Comments: 1. This device can select ions or masses on the principle of their oscillating frequency in a static "potential well" where the ions pass several times across the same rf field tuned to their oscillation. The particles gain energy on each passage. The initial velocity of the particles is not of primary importance; thus, there may be more energy spread in the ion source. This is not so in most mass spectrometers. No high mechanical precision is required, also the resolving power may be varied by electrical means. A phenomenon such as "harmonic mass" appears but can be readily eliminated. II. The most important fact garnered from this new idea is that no magnetics are used. The implications are seen to be many here: This instrument could be made comparatively portable and perhaps small enough to probe likely crevasses for leaks by detecting the ions somehow produced. It seems that this idea of Bierman's could be used profitably to detect masses and subsequently leak rates. However, what could be used to ionize the leaking gas that would be small in size?

Boyd, R. L. F., and D. Morris. "A Radio Frequency Probe for the Mass-Spectrometric Analysis of Ion Concentrations," Proc. Phys. Soc. (London) A, 68, Pt. 1, 1-10, Jan., 1955.

Describes the development, and analyses the action, of a versatile probe which can readily be moved radially into and out of a discharge tube and which is able to distinguish between ions (positive or negative) of various masses. With this instrument it

is therefore now possible to make probe studies on a particular species of ion in the presence of others. Basically the instrument is a very small 12-stage linear accelerator which discriminates in favour of ions of a particular mass-charge ratio passing through its sampling orifice. It is of high sampling efficiency and this together with its mobility and the absence of a magnetic field gives it certain pronounced advantages over the magnetic method of analysis.

Bradley, R. S. "A Thermistor McLeod Gauge for a Pressure Range, $1\text{-}10^{7}$ mm of Mercury," J. Sci. Instrum., 31, 129-30, April, 1954.

A gauge capable of reading pressures of non-condensable gases in the range $1\text{-}10^{-7}$ mm Hg has been made by sealing a thermistor bead into the end of the closed capillary of a McLeod gauge, the thermistor acting as a thermal conductivity manometer of the Pirani type.

Brubaker, W.M., and V. Wouk. "Frequency Modulated Oscillator for Leak Hunting," Rev. Sci. Instrum., 17, 97-8, March, 1946.

A circuit for converting a variable d.c.,voltage signal into a frequency modulation of an audio-oscillation is described. The circuit can be used for general monitoring purposes, but this article confines the description to its use in conjunction with an ionization gauge for leak hunting in vacuum systems. The oscillator enables one operator to hunt leaks more rapidly and effectively than two men calling out meter readings. Leaks representing pressure rises of less than 4×10^{-8} mm of Hg have been found with an ionization gauge and this oscillator.

Burch, Darrel E., David Gryvnak, and Dudley Williams. "Infrared Absorption by Carbon Dioxide," Scientific Report No. 2, Contract AF 19 (604) 2633, Jan., 1961.

Descriptors: *Carbon dioxide, *Gases, *Nitrogen, Infrared radiation, Absorption, Infrared spectroscopy, Measurement, Pressure, *Atmosphere.

The infrared absorption of carbon dioxide was investigated. Samples consisting of carbon dioxide alone, and in binary mixtures with nitrogen, were investigated over wide ranges of absorber concentration w and total pressure P. From the absorption by each

band or spectral interval, it was possible to determine either the total absorption or the mean fractional absorption for each sample. Curves are presented which show the total absorption of the CO_2 bands at 1/3716, 1/3609, 1/2350, 1/1064, and 1/961 cm for various values of absorber concentration as a function of equivalent pressure P sub e, which is a parameter that includes the total pressure P and a small additional term proportional to the partial pressure of the absorbing gas. Absorption curves for the 1/1064 and 1/961 cm bands as well as for the 1/875 to 1/495 cm region, were obtained for different samples of CO_2 at temperatures from ambient to approximately 70 C. For the temperature range covered it is possible to predict the increase in total absorption of the 1/1064 and 1/961 cm bands by use of an elementary function which is based on the calculated increase in population of the lower energy level giving rise to the absorption.

Catlin, Franklin. "Fluorescent Method Detects Leaks in Process Vessels," Chem. Met. Eng., 50, 116, 1943.

Leaks are detected by coating the surface of the vessel with a fluorescent penetrant and inspecting the opposite surface with a near-ultraviolet lamp.

Coggleshall, N.D., and E. L. Saier. "Analyses of Mixtures of Light Gases by Infra-Red Absorption," J. Appl. Phys., 17, 450-6, June, 1946.

A discussion is given of the application of infra-red absorption methods of analysis for light gases which do not obey Beer's law of absorption due to pressure broadening. The method depends upon the nature and intensity of the pressure broadening effect of the different components in the sample upon each other. Data are presented showing the nature of some of these effects and illustrating the accuracy obtainable for certain types of analyses. The instrumentation used in routine gas analyses by infra-red is described.

Conn, G. K. T., and H. Daglish. "A Simple Thermionic Vacuum Gauge," J. Sci. Instrum., 31, 95-6, March, 1954.

A description is given of a gauge which, because of the form of the anode, is very easy to construct. The release of photoelectrons which limits the use of a conventional thermionic gauge at low

pressures is surmounted by using a single wire as the ion collector. At pressures of the order of 10^{-4} mm Hg or less the instrument is linear and has a sensitivity of about 13μ A per μ pressure per mA anode current.

Conn, G. K. T., and H. N. Daglish. "Cold Cathode Ionisation Gauges for the Measurement of Low Pressures," Vacuum, 3, 24-34, Aug., 1954.

Cold cathode ionization gauges in which the discharge is maintained in an axial magnetic field provide a convenient and rapid means of measuring pressure in the range 10^{-2} to 10^{-7}mm Hg. These instruments are simple to make and operate and it is virtually impossible to damage them. With such a gauge, measurement of pressure is effected by a robust pivotmeter which measures a current of ions formed by electron bombardment. Primary electrons are released by cold emission from a pair of cathodes and many secondary electrons are generated by the ions themselves. The magnetic field causes the electrons to move in tight spirals to and from between the two cathode plates. In this way the number of ions per electron, between initial emission and eventual escape from the discharge to the anode, is much increased . A review is given of the construction, characteristics and performance of various types of cold cathode gauge. The influence is reported of size and shape of the electrodes, of applied voltage, of magnetic field and of the nature of the gas. Reference is made to the performance at relatively high pressures (10^{-2} to 10^{-7} mm Hg). Attention is drawn to the generation of radio frequencies by cold cathode gauges.

Cook, D. B., and C. J. Danby. "Simple Diaphragm Micromanometer," J. Sci. Instrum., 30, 7, 238-40, July, 1953.

Vacuum gage covering pressure range 1 to 100 microns with accuracy of better than 0.5 microns; movement of diaphragm is followed by measurement of capacity change; electronic circuit involves only single tube; gage is suitable for measurement of mass spectrometer sample inlet pressures; circuit diagrams.

Crampton, D. H., and Carl Winnefeld. "Leak Detection of Refrigerants," Refrig. Eng., 55, 26, 261-3, 1948.

Methods now in use are the soap bubble test, sound effect, chem. swabs, halide torch, and the color tracer method. The color

tracer method is one of the most dependable. A red oil-soluble dye dissolved in oil of medium viscosity is introduced into the refrigerating system and leaks are detected by observation on the distribution of the color.

Crouthamel, C. E., and H. Diehl. "Gas Analysis Apparatus Employing the Velocity of Sound," Analyt. Chem., 30, 515-20, June, 1948.

A brass tube of fixed length containing the gas has a transmitter (1-3 kc/s) at one end and a microphone at the other. Change in gas composition causes departure from resonance conditions, and change in microphone output. An amplifier feeds a meter calibrated directly in terms of the composition, and a warning can be given when one component exceeds a preset proportion.

Davis, A. D. "Thermistor Detectors in Gas Chromatography," J. Appl. Chem. (London), 8, Pt. 3, 183-6, 1958.

Details are given of a simple and easily constructed instrument, employing a thermistor detector, for work on gas chromatograph. Necessary precautions and conditions for use of a thermistor detector are outlined.

Dibeler, V. H., and F. Cordero. U. S. Bur. Stand. J. Res., 46, 1-4, Jan., 1951.

A diaphragm-type micromanometer is described that is capable of measuring pressures in the range of 1 to 100 microns with a sensitivity of about 0.1 micron on the 50 micron scale. The displacement of a thin diaphragm is measured by the change in mutual inductance of two coils mounted above the center of the diaphragm. Like other manometers, this does not appear to apply.

Drexler, Milton, and Myron Barchas. "Chemo-Electrical Sensing Device," Unclassified Report, RADC TR 61-177, June, 1961.

The feasibility of utilizing gas adsorption phenomena in the identification and quantitative determination of various gaseous materials was investigated. Factors influencing the performance of the various components of an experimental gas analyzer based on the measurement of characteristic adsorption energies are discussed. Instrumentation specifications included ability to detect and

measure concentrations of 10 ppm or less of NO, NO2, N203, HCl, HF and 03 in air, irrespective of the mixture involved. The system involved passing the air for analysis through a sensing cell which was to measure the heat of adsor ption of the contaminant on an adsorbent-coated thermistor. On the basis of experimental and theoretical analysis it was concluded that a gas sensing device based on the principle of differential heats of adsorption is not feasible.

Florio, F. "Automatic Safety Devices for Rocket Propelled Aircraft," Jet Propulsion, 25, 235-6, 1955.

Described is a unique liquid-leak-detecting device for use with HNO_3 rockets. NaOH is wrapped in a gum-based coating which is destroyed by liquid HNO_3. Thus an electrolytic connection is made between 2 Ag-sheet electrodes, thus permitting a current to flow to a warning light or relay. The small unit is about 1 in. in diameter, and one drop of HNO_3 establishes a current of 150 ma in 0.2 sec. Insulation of the bag is about 10 megatrons, but after lengthy exposure to acid vapors the insulation value does not drop below 1 megatron.

Garton, W. R. S., M. S. W. Webb, and P. C. Wildy. "The Application of Vacuum Ultra-Violet Techniques to the Continuous Monitoring of Trace Concentrations of Water in Several Gases," J. Sci. Instrum., 34, 12, 498-500, Dec., 1957.

A method for the determination of water in several gases is outlined, in which use is made of the absorption bands of the water molecule in the 1200 A region. A 20 Mc/s electrodeless discharge in hydrogen excites the 1216 A Lyman α line very strongly and relatively free from neighbouring lines and bands. The radiation passes through the absorption cell, closed at both ends by LiF windows, on to the W cathode of a photomultiplier, which is insensitive to light of wavelength longer than 1400 A. Contains a description of the method and a discussion of the apparatus.

Geller, R. "Leak Detection of Vessels under High Pressure with a Helium Mass Spectrograph," Vide, 12, 71, 398-406, 1957.

Two methods are discussed. According to the 1st, the vessel is filled with He at more than 1 atm. pressure and tested with a moving probe connected to the leak detector. Calcu. and estimates are made

of the sensitivity of the method and the min. discernible leak. According to the 2nd method, a sealed small vessel is first introduced into a recipient under high pressure and then placed in an evacuated container connected to a leak detector. The sensitivity of this method is equally discussed.

Gemant, A. "Tracer Diffusion in the Ground in Radioactive Leak Location," J. Appl. Phys., 24, 93-5, Jan., 1953.

Diffusion in ground of radioactive tracer gas, used for locating leaks in buried pipes, has been calculated, and results presented in graphs; how information can be used to obtain amount of radioactivity needed in test for both beta emitters (C-14 in carbon monoxide) and gamma emitters (Br-82 in methyl bromide).

Gemant, A., Edward Hines, and E. L. Alexanderson. "Leak Location by Radioactive Gases in Buried Pipes," J. Appl. Phys., 22, 460-4, 1951.

C^{14}-labeled CO and Rn were used as tracers; CO_2 was not used because it is readily absorbed by the soil. The principles of the method are described. Suitable techniques for prepg. and detecting the tracer gas were worked out. The spreading of a column of tracer gas in a pipe is calcd. Leaks are located on two small-scale installations.

Gilpin, Wm. V. "Southern Union Gas Company Uses New Device for Finding and Locating Gas Leaks," Am. Gas J., 180, 5, 28-9, 1954.

Description and operation of a successful bubble-type leak detector for leaks in gas service lines. The location of the leak can be detd. by addn. of a leak-detecting chemical, such as Me chloride, to the unit. The air picks up a little of the chemical, and the presence of the chemical at the location of the leak can be detected by use of a halide lamp and sniffer tube.

Greenough, M.L., and W. E. Williams. U.S. Bureau of Stand. J. of Res., 46, 5-6.

An instrument has been developed for indicating the position of thin pressure sensitive diaphragms. The electronic instrument gives full scale indication for motions of less than 5×10^{-4} inches. Five tubes are incorporated in a circuit based upon the principle of mutual inductance micrometer for the detection of diaphragm motion.

Griffiths, J. H., and C. Phillips. J. Chem. Soc., 3446-53, 1954.

The method involved is the chromatography of gas and vapors and applications of the surface potential detector. The device consists of two identical metal plates connected by a conductor. One plate can be made to vibrate by an oscillator. If the plates are different in surface treatment, an alternating emf will occur when the plate is vibrated which can be measured by applying a bias emf to one of the plates. If surfaces are charged by exposure to an adsorbable vapor, the potential will be altered and this changed can be amplified and read. Here they use N as a vehicle for the gases to be analyzed. The apparatus features high sensitivity; i.e., 4 p.p.m. of ethyl oxalate in N_2 gives a signal of 7.5 mv.

Guthrie, A. "Leak Detectors for Industrial Vacuum Systems," Electronics, 23, 96-101, Sept., 1960.

The main desiderata for such detectors are: (1) capability of measuring total leakage and isolating individual leaks; (2) rapidity in response; (3) high sensitivity; (4) adaptability to any vacuum system without loss of vacuum; (5) relative simplicity and low cost; and (6) selectivity. Of eight tabulated methods, the He leak detector is generally the most useful for rapid operation. It is based on the vacuum analyser, a form of small-radius mass spectrometer. It consists of a non-magnetic tank, supported by and connected to the vacuum line, and in it are mounted a cold-cathode positive-ion source, a collector, and a magnet system. A sweep voltage superposed on the ion-accelerating voltage sweeps the He mass peak across the collector slot. The collector is connected through an amplifier chain to (1) a c.r.o. with horizontal sweep in phase with the other sweep voltage, (2) a leakage-detecting meter following a filter for removing all frequencies except the sweep frequency, and (3) a 1 kc/s "squealer" circuit. Leak sensitivity $\simeq$ 1 part of He in 2×10^5 parts of air and is limited by noise in the amplifier, stray frequency pick-up, and ion background. Methods of determining sensitivity are described and response time discussed. The whole procedure of searching for leaks and their subsequent repair is explained in detail.

Hafner, A. "A Portable Thermistor-Bridge Gas Leak Detector," NRL Report 5647, 1-19, July, 1961.

A new portable gas leak detector useful for locating leaks in pressurized systems containing any gas having a thermal conductivity different from that of air is described. The detector is accoupled and hence does not respond to the gas concentration in the ambient air but only to the change in concentration encountered in the vicinity of the leak. Investigations and experiments led to a choice of a thermistor bridge in which optimum response time and minimum sensitivity to gas flow rate variations is obtained by using thermistors shielded from the turbulence of the direct gas flow by glass envelopes but with a hole in each envelope to admit the gas by diffusion. Care was taken in the circuit design to prevent blocking of the high-gain amplifier by noise. Sensitivity of the gas leak detector was checked against a calibrated leak of Freon-12, and the device proved to be useful in locating Freon F-12 leaks of as small as 2 ounces per year = 1.3×10^{-7} SCIm. It was used with marked success during submarine operation.

Heath, H. "Initial Experience with the Palladium-Ion Gauge Method of Vacuum Testing," Gt. Britain, Capenhurst Wks., Capenhurst, Ches., England, Oct. 21, 1952.

A thin heated Pd sheet between an ion gage and a vacuum chamber allows H_2 exclusively to enter the ion gage. Thus, a leak probed with H_2 shows up even with heavy outgassing and with short pumping times. Operation of and operating experience with such a test arrangement are described.

Hershberger, Wm. D. "Gas Analysis by Use of Microwaves," U.S. 2,792,548, May 14, 1957.

The analysis of gases is carried out by detn. of selective absorption, variation in dielec. const. at const. pressure, or as a function of pressure, for mm. or cm. electromagnetic waves. The procedure has been applied to the monitoring of the production of NH, by the Haber process. The effect of gas pressure on microwave absorption is detailed and derived so that an optimum pressure or pressure range can be selected for the particular analysis.

Hobson, A., and R. H. Kay. "Two Designs for a Paramagnetic Oxygen Meter," J. Sci. Instrum., 33, 5, 176-81, May, 1956.

A robust oxygen detector capable of giving direct readings on a meter or recorder has many applications in physiology or in the control of chemical processes. Physical methods at present available commercially have characteristics which often make them unsuitable for particular applications. This paper describes two separate but parallel investigations of the design of an oxygen meter depending on the paramagnetism of oxygen and the change of its susceptibility with temperature. One instrument covers 0-100% oxygen concentrations at or near atmospheric pressure, the other 90-170 mm of mercury partial pressure of oxygen in total ambient pressures of about 600-900 mm of mercury.

Huguenard, M. E. "Electrical Method for the Instantaneous Determination of Traces of Gas in Air," Compt. Rend., 213, 21-3, 1941.

Changes in the compn. of air are detd. by changes in the resistance of a Pt wire heated to 1200-1300°. CO, H_2S, H_2, NH_3, alc., ether and illuminating gas cause exothermal reactions and thereby increase the resistance. CO_2, H_2O and CCl_4 dissoc., cool the wire and thus decrease the resistance. The nature of a foreign gas or of a mixt. can be detd. from its behavior on a Pt wire when heated to various temps.

Hurst, W. "Recording Sensitive Differential Manometer," Rev. Sci. Instrum., 12, 265-68, May, 1941.

A sensitive differential manometer used in the recording of small differential pressures is described. This instrument responds to pressure differences as small as 0.00003 cm of Hg and as rapid as 80 c./s. The moving element is a rectangular glass mirror 0.010 in. thick and approx. 0.060 in. x 0.080 in., supported on a glass shaft 0.007 in. in dia. Motion is supplied by a thin rubber diaphragm approx. 0.003 in. thick. Included in recordings made with the aid of this instrument are wave forms originating in the human neck and finger, also pressure changes in a glass cell enclosing a tracheal breathing insect.

Johansson, Gillis. "Gas Analysis by Use of Microwaves," Analytical Chemistry, 34, 8, 914-6, July, 1962.

The properties of microwave circuits permit highly accurate measurements of dielectric constants. A simple arrangement of two cavities in series can be used for very sensitive relative measurements. Traces of one gas in another gas can be detected if the dielectric constants differ, which is generally the case. The response law is derived and verified experimentally. The influence of selective absorption and dielectric loss is discussed. The device will find application as a detector in gas chromatography, especially at high temperatures. A differential type detector employing this principle could be designed.

Kent, T. B. "A Hydrogen Leak Detector Using a Charcoal Trap," J. Sci. Instrum., 32, 132, 1955.

1. Method - change in pressure. 2. Detector- Pirani gauge. 3. Detected - a change in pressure caused by inducing hydrogen into the system through the leak. 4. Kind and size - from atmosphere to vacuum - any size. 5. Sensitivity - smallest detectable leak $\sim 4 \times 10^{-4}$ 1μ/sec. 6. Instrumentation - Pirani gauge, galvanometer, etc. 7. Ease and speed - system must be pumped down first. System is pumped down and enclosed in a hydrogen atmosphere. The charcoal trap readily absorbs most atmospheric gases but will pass nearly all of the hydrogen, thus ensuring that only the hydrogen gets into the system. If there is a leak, then, the hydrogen is induced into the system and the Pirani gauge indicates the resulting change in pressure.

King, Robert W., Jr. "Thermistor as a Flowmeter," Res. Div., New York University, Oct., 1951.

Thermistors - uses 2. Meters, Flow - Electromagnetic.

Kittaka, Shigeyoshi, Kozo Yamagata, Toru Sukegawa, and Tamio Sakata. "The Use of Thermistors for the Analysis of Gas," Ōyō Butsuri, 22, 119-20, 1953.

The change of thermal cond. λ due to the change in compn. of a mixt. of gases (CO_2 and H_2) is measured by the change of temp. t of a thermistor placed at the center of the vessel. $dt/d\lambda$ is given by the Chapman equation (C.A. 34, 1240^4). The reproducibility and the deviation from the linearity at high concn. in air are examd.

Krumbein, A. D., F. A. Grant, and A. L. Ward. "A Light Absorption Method for the Quantitative Determination of Small Changes in Chlorine Concentration, Letter in J. Opt. Soc. Amer., 42, 277-8, April, 1952.

An illustrated description of a photoelectric absorptiometric apparatus for measuring small changes in the concentration of the Cl component in the counter filling, using a simplified Beer's law expression $I = I_0(1 - \alpha cd)$ which has been found to be valid up to a Cl pressure of 3 mm Hg. A method to measure changes in Cl concentration in Halogen counter tubes by absorption of ultra violet light.

Lefort, M. "Utilization of the Ionization Produced by Alpha Rays for the Microanalysis of Gases," J. Phys. Radium, 17, 2, 164-5, Febr., 1956.

The method depends on the fact that for a given pressure the ionization current in an alphatron gauge depends on the nature of the gas.

Lehrer, E., and E. Ebbinghaus. "An Apparatus for Determining Oxygen Magnetically in Gas Mixtures," Z. Angew. Phys., 2, 1, 20-4, 1950.

Constructional and operational features are given of a recording apparatus for estimating oxygen in technical gas mixtures.

Leont'ev, N. I., and Yu. K. Udovichenko. "Omegatron," English translation of Pribory i Tekhnika Eksperimenta, 1, 105-6, Jan.-Feb., 1959.

Mass analyzer for measurements in region of light masses; principle of operation of instrument, which is highly sensitive due to absence of collimating slots, is based on cyclotron resonance of ions; accuracy for determining isotope content of normal neon is plus or minus 5%.

Lloyd, J. T. "An Audible Vacuum-Leak Indicator," J. Sci. Instrum., 27, 76-7, March, 1950.

An apparatus is described which is suitable for indicating pressure changes over the range from 10 mm to less than 0.1 mm of mercury. It employs a two-electrode discharge tube whose electrical characteristics depend on the nature and pressure of the gas it

contains. If such a tube is inserted in a vacuum line and used as a variable factor in a simple relaxation oxcillator circuit, the oscillation frequency, made audible by a loudspeaker, will serve as an indicator of the discharge conditions. The device is stated to be particularly useful for testing Geiger-Mueller counters using a two-stage rotary pump.

Loevinger, R., and H. Guthrie. "Leak Detection Instruments and Techniques," Vol. I, Chap. V of AECD-2405 (BP-93) and Decl, Dec. 6, 1948, for publication in NNES.

The locating and repairing of leaks in vacuum systems is probably the most troublesome aspect of high vacuum technique. In this chapter is given first a rough method of estimating the flow of various gases through small capillary holes, then a description of all the successful methods of leak detection normally available and finally an indication of the method of formulating quantitatively the performance of leak detection techniques. Information is submitted concerning the flow characteristics of small capillaries, methods of leak detection, pump-outs, the vacuum analyzer, the helium leak detector, general leak hunting procedure, repairing of leaks, outgassing, virtual leaks, and theory of leak detection. 19 references.

Loevinger, R., T. A. Chubb, and G. W. Monk. "Leak Detector," To U. S. Atomic Energy Commission, U. S. Patent 2,727,995, Dec. 20, 1955.

A leak detector is described using He or other appropriate gas for the detection of leaks in tanks, housing, or other equipment where it is necessary to know the effectiveness of the seal or air tightness thereof in connection with maintaining vacuum or pressures therein. If He is used as the gas, it can only enter the detector through the sealed chamber to be tested thus providing an indication of any leak. Any such He molecules entering the detector are ionized by means of a cold cathode ionization chamber. The ions are then accelerated into a region of lower pressure where they are caused to follow arcuate paths thus providing an ion beam. A charged electrode is displaced from the ionization chamber along the arc of travel of the ions at substantially the focal point of the beam in order to suppress the ions of low energy content. A collector is disposed beyond the focal point on the arc of travel of the ions for collecting the high energy ions. The important

feature of this invention is the provision of the suppressor electrode which operates to permit only the He ions to reach the collector.

Lortie, Y. "Utilization of Thermistors as Vacuum Gauges," J. Phys. Radium, 16, 4, 317-20, April, 1955.

A study of the use of thermistors in Pirani-type gauges in the pressure range 1 to 10^{-3} mm of Hg. Uses a Wheatstone bridge and keeps the glass envelope containing the thermistor at constant temperature.

Luck, C. P. "Membrane Manometer with Secondary Air Transmission," Journ. of Sci. Instrum., 28, 173-6, June, 1951.

This is an instrument designed to take precise blood pressure readings and to record them.

Madan, M. P. "Simple Bridge Method for the Measurement of Thermal Conductivity of Gases and Gas Mixtures," J. Franklin Inst., 263, 3, 207-12, Mar., 1957.

A simple bridge (hot-wire) method for the measurement of thermal conductivity of gases or gaseous mixtures is discussed and its design developed. A rigorous mathematical theory is applied to calculate the various corrections and to obtain the value of the thermal conductivity in terms of the conveniently measured quantities.

Magee, J. B., and M. Crain. "Recording Microwave Hydrometer," Rev. Sci. Instrum., 29, 1, 51-4, Jan., 1958.

Describes a rapid response microwave hygrometer for continously recording the water vapour pressure of atmospheric air over a wide ambient range. The principle employed involves the measurement by means of a cavity resonator of the contribution of water vapour to the refractive index of atmospheric air. The device described also has potential application to the continuous measurement of the degree of contamination of one gas (or of gas mixtures) to another gas (or gases).

Martin, A. E. "The Sonic-Gas Analyzer," Nature (London), 178, 407-8, August 25, 1956.

Refers to the successful development of a commercial form of sonic gas analyzer by Dawes, Walton, Lawley and Mounfield. The principle of operation involves the use of two equal tubes (length d) each fitted with a deaf-aid earpiece at each end. At end A of each tube the "earpiece" is used as a sound source (3000 c/s) while at the opposite end B of each tube the earpiece is used as a microphone. By electronic techniques it is easily possible to measure the phase difference of the sound arriving at the two receivers $B_1 + B_2$. This is zero when the gases filling the tubes are the same but becomes finite and measurable if one gas is heavier than the other.

Medlock, R. S. "Oxygen Analysis Based on Its Paramagnetic Properties," Trans. Instrum. Meas. Conf., Stockholm, 102-6, 1950.

The "magnetic wind" is described, by means of which the O_2 may be measured by its paramagnetic property. An improved instrument has been devised and its performance mathematically determined. One recorder had an accuracy of $\pm$ 0.05% O_2 in a range of 0-5% O_2, with a 10 in. scale.

Miettinen, Jorma K. "Gas Chromatography - a Review," Suomen Kemeistilehti 31A, 149-74, 1958.

48 references listed here.

Millar, A. R. "Theory and Design of an Acoustic Pressure Gauge," Atomic Energy Res. Establ. (Harwell) Memo. GP/190, 1-11, 1956.

In the pressure range 10^{-2} to 10^{3} mm mercury a number of well-established types of gauge are in use. These include manometers (mercury or oil), McLeod gauges, Pirani gauges, and comparison devices using a mechanical diaphragm. Each of these types has its own limitations in convenience, accuracy, or working range. A type of gauge is proposed here in which these limitations could be less serious, and which will cover the whole of the above range. Operation of the instrument depends on the amplitude of received signal at the end of an "acoustic transmission line", which is simply a tube containing gas at the pressure to be measured. A simple form of the gauge has already been constructed, which works well for air; design proposals are given for a gauge of general applicability.

Miller, R. D., and M. B. Russell. "A Continuous Analysis of Gas Streams," Anal. Chem., 21, 773-7, July, 1949.

A continuous analysis of gas streams is provided by the absorption of bands in the infra-red by heteroatomic gas molecules. The analyzer was made of brass tubing and the appropriate cells. A picture of the instrument shows a boxlike and rather cumbersome object. It does not appear suitable for NASA.

Minter, Clarke C. "A New Thermal Conductivity Leak Detector and Its Applications," Proj. NS-131-004, Naval Res. Lab., Apr. 25, 1958.

This report points out the reasons for the failure of the Pirani tube as a sensitive leak detector and describes a simple thermal conductivity apparatus of special design having several advantages over the mass spectrometer so widely used for this purpose. Procedures are described for using the new apparatus to locate leaks in vacuum or pressure systems using hydrogen or helium as probe gas. Experiments to determine the sensitivity of the apparatus toward small changes in concentration of freon in air are described, and it has been concluded that leaks in refrigeration equipment can be located even when appreciable concentrations of freon are present in the ambient air.

Minter, C. C., S. F. Anderson, and R. W. Dolk. "The NRL Model E-5753 Leak Detector, Report 18, Nav. Res. Lab., Jan., 1961.

A 21-pound portable Freon leak detector has been developed. Air from the vicinity of a probe is pumped through tubing so as to first pass two of the four cells of a thermal conductivity bridge and then through a length of tubing around to the other pair of cells. Thus, if the probe is moved past a leak, the bit of Freon-containing air unbalances the bridge first in one direction and then in the other. These unbalances cause the successive flashing of a red light and a white light in the Lucite probe head, where the operator's attention is focused. Since the air flows past one end of each conductivity cell and not through it, a lag time occurs before the sample diffuses into the cell and reaches the filament; this lag time is predominant in producing a total lag time of at least 3 to 4 seconds. Bridge excitation is by a regulated ac power supply, and the ac output signal when the bridge is out of balance goes to a five-stage RC-coupled transistorized amplifier. Printed circuit and plug-in construction facilitates circuit checking and amplifier replacement. While the indicator gives a clear response

for a Freon concentration of 4 to 5 ppm, such sensitivity is not needed under service conditions, where detection of the signal produced by changing the concentration of Freon by 50 ppm is satisfactory in most cases.

Minter, C. C. "Thermal Conductivity Leak Detector," R. Sci. Instrum., 29, 793, 1958.

Method - electronic. A metal bridge block consisting of 4 cells with filaments was connected to form a Wheatstone bridge. A change in thermal conductivity was detected. Kind and size: a leak from atmosphere to vacuum. Sensitivity: $\sim/ \times 10^{-9}$ atm. cc/sec. Believed utlimately to be better than mass spectrometer. Instrumentation: potentiometers, d.c. constant voltage source, sensitive μ ammeter. Seems to be relatively easy to use. The bridge block is connected between vessel being tested and pump in such a way that the gases from vessel must pass through both sides of bridge block; that is, through all four cells. The bridge will be unbalanced due to drop in pressure in the gas stream which will cause the filaments in the cells nearest the pump to have a slightly higher resistance than those nearest leak. The bridge is then balanced, after which any probe gas coming to bridge block will cause the bridge to be unbalanced and the indicator on the μ ammeter will show a deflection. It is supposed to be easier to use than mass spectrometer. No mention is made of nature of probe gas.

Minter, C. C. "Vacuum Leak Testing with Liquids," R. Sci. Instrum., 31, 458, 1960.

The method is thermal conductivity bridge. The presence of liquids such as water, heavy water, alcohols, and acetone are detected. There are small leaks in the vacuum system. Sensitivity is to water vapor - 33 mv/μ of vapor. Instrumentation: thermal conductivity bridge, moving coil indicator. If a drop of water, or some other liquid having a high latent heat of vaporization, is put on the leak the thermal conductivity increases inordinately.

Nelson, H. "The Hydrogen Gauge - an Ultra-Sensitive Device for Location of Air Leaks in Vacuum-Device Envelopes," Rev. Sci. Instrum., 16, 273-5, Oct., 1945.

A sealed-off, highly evacuated ionization gauge has a section of its

envelope made of thin Pd sheet which, when heated, is highly permeable to hydrogen. The gauge is attached to the manifold of a vacuum system with the Pd section isolating it from the vacuum. When the manifold or any vacuum device connected to it is probed with hydrogen, leaks in the system are indicated by an increase in the ion current of the gauge. This new technique, because it utilizes a sealed-off, highly evacuated gauge which responds only to hydrogen and is unaffected by other residual gases and vapours, provides a simple but sensitive test for very small leaks.

O'Bryan, H. M. "Rayleigh Interferometers for Gas Analysis," J. Opt. Soc. Amer., 34, 774, Dec., 1944.

Describes modifications of the Rayleigh interferometer for measuring refractive indices of gases. One is the use of a four-compartment cell, giving twice the sensitivity without increase in overall length. Another is the use of auto-collimation, in combination with a four-compartment cell, in a portable instrument for measuring concentration of toxic gases in industrial atmospheres.

Ochert, N. "Leak-Detection Practice with Particular Reference to the H_2-Pd Method," Vacuum, 2, 125-36, 1952.

Leaks to 10^{-2} lusec can be detected by a Pirani gage by using H_2 as test gas. Leaks to 10^{-5} lusec can be found by a Pd barrier ionization gage. In this gage the anode consists of a Kovar tube, closed by a Pb cap, which is heated to incandescence by electron bombardment. Difficulties of the method due to cracking of hydrocarbons and O_2 combination with H_2 are discussed. A leak-detection plant contg. both units and leak-testing techniques in mass production are described. Leak rates can be expressed quantitatively if the app. is calibrated with the aid of a calibrated reference leak, the construction of which is described.

Pauling, L., R. E. Wood, and J. H. Sturdivant. "An Instrument for Determining the Partial Pressure of Oxygen in Gas," Science, 103, 338, March 15, 1946.

The operation depends on the fact that oxygen has a much higher magnetic susceptibility than any other gas. The force on a test body surrounded by the gas in an inhomogeneous field is measured by means of a torsion balance. The precision depends on the range of pressures for which it is to be used; e.g., it is $\pm$ 1 mm for a range 0.180 mm of Hg.

Perkins, James C., Jr. "An Investigation to Determine a Practical, Precise and Reliable Method for Detecting Leaks in Rubber Handwear," Tech. Rept. R-6; FEA MRS 6001, March, 1960.

This study was conducted to develop a practical,precise,and reliable method of inspecting rubber protective handwear under field test conditions. Three methods were tested. They are (1) high definition fluoroscopy, (2) halogen detector, and (3) infrared nitrous oxide analyzer. Each of these methods employs a tracer gas and a detector or sensing element. The leak detector tracer gas (halogen) was found to be the most satisfactory method for making determinations of rubber handwear serviceability.

Philips, N. V. "Detecting Traces of Foreign Gases, etc., in the Atmosphere by Means of a Surface on which a Reaction is Going on that is Influenced by These Substances," Dutch, 54, 716, June 15, 1947.

Reactions under the influence of a catalyzer (Pt., ferric oxide, ferments, or enzymes) can be interferred with by catalyzer poisons such as As_2O_3, CO, HCN, H_2S, or org. S or As compounds. This is shown by changes in the caloric effect of the reaction, made visible by elec. app.

Phillips, G. "An Electronic Method of Detecting Impurities in the Air," J. Sci. Instrum., 28, 342-7, Oct., 1951.

Impurity concentrations in clean dry air of one part in 10^7 by weight of polar vapours or of one part in 10^4 by weight of non-polar vapours were measured in terms of changes in surface potential of a prepared plate. Changes of potential of the order of 10μ V were measured using a vibrating condenser technique and a selective phase-sensitive amplifier. The results suggest that while the adsorption is caused mainly by electrostatic attraction between the dipole and its induced image, a small contribution is also caused by van der Waals attractive forces. The possibilities of obtaining selective adsorption, and some of the practical applications, are discussed briefly.

Poole, R. "The Design, Testing and Calibration of a Combustible-Gas Detector," J. Instn. Elect. Engrs., Pt. II, 95, 258-74, June, 1948.

This article is concerned with the design, testing, and calibration

problems that must be taken into account in combustible gas detectors. Research into different types of detecting filaments is discussed, particularly platinum, and poladium. The article is concerned with detection of hydrocarbon gases. The only particular detector discussed is a portable gas detector of the indicating type employing the catalytic filaments cell and single gas chamber. It is for measuring the amount of gas (hydrocarbon) in the air and not for locating sources. It measures concentrations as low as 1/50 of 1% by volume. This is a general discussion of detecting problems. Any detectors mentioned (not in detail) are used in industry and are rather common.

Pressey, D. C. "Temperature-Stable, Capacitance Pressure Gauges," J. Sci. Instrum., 30, 20-4, Jan., 1953.

A brief theoretical treatment is given of the effect of temperature changes on capacitance pressure gauges. Its use in the design of a sensitive differential pressure gauge of range 0-10 mm of water, having a temperature coefficient of capacitance of only -100 p.p.m./^{o}C, is illustrated.

Reinders, M. E., J. Schutten, and J. Kistemaker. "Leak Detection with a Mass Spectrometer using Hydrogen Gas," Appl. Sci. Res., B2, 1, 66-70, 1951.

The minimum detectable leak rate into the ion source (pumping speed 1 litre/sec) was found to be 0.28 mm^3 h (N.T.P.) for H_2^+, compared with 1 mm^3/day for He. 1 part of hydrogen was detectable in 10^4 parts of air, and 1 of helium in $5\text{-}10^4$ of air. A Philips ion gauge just detected 50 mm^3/hr using ether vapour, so that the spectrometer may be used with advantage on hydrogen when helium is not available. Figures are given for other probe gases.

Reis, T. "Note Concerning the Detection of Leaks by Means of a Pirani Gauge," Cahiers de Phys., 46, 59-62, Nov., 1953.

The use of a Pirani gauge as a manometer for evacuated systems is based on the change in temperature of a heated metal filament, which varies with the pressure and the thermal conductivity of the surrounding gas. The gauge can be calibrated, for a given gas, by introducing the gas into the system through an artificial leak. Hydrogen, owing to its high thermal conductivity, was found to be most suitable. Details of apparatus, calibration and two examples of leak detection at different pressures, are given.

Romand, Jacques, Vladimir Schwetzoff, and Boris Vodar. "Application of Optical Absorption in the Far Ultraviolet to Leak Detection in Vacuum Apparatus and to the Measurement of Low Pressures," Vide, Le 6, 1046-51, July-Sept., 1951.

An apparatus measuring absorption in the Schumann region (1850 to 1200 A) is illustrated and described. The use of the equipment in measuring air pressures down to 5×10^{-4} mm Hg and leaks, using benzene as a tracer, of 5×10^{-9} liter/sec is reported.

Romand, Jacques. "Possibilities for the Utilization of Schumann Ultraviolet Absorption Spectra for the Detection and Determination of Gases, Application to the Detection of Leaks in Vacuum Apparatus, and the Measurement of Low Pressures," Congr. Groupement Avance. Méthodes Anal. Spectrograph, Produits Met., 14, 201-19, 1951.

Optical absorption in the region 2000-1250 A. provides a means of measuring rapidly and precisely the density of air in a wind tunnel since O absorbs strongly around 1450 A. The method does not depend on the rate of gas flow and can provide a measure of the mean pressure perpendicular to the air streams without perturbing them. The strong absorption of benzene at 1790 A. can be used to search for leaks. The photomultiplier used has a Pt cathode for which the threshold is about 2000 A. The app. consists of a H lamp with a fluorite window, and the receiver, whose window is blown from quartz of high purity. The source and receiver are connected by a brass tube to which are attached the pumping station and the object to be tested. The amplifier used has a gain of 2×10^{8} and max. deflection of the microammeter occurs for 50 microamperes. The measurable pressure range is varied by changing the length of brass tube. For the limits 0-400 microamperes the respective pressure limits are $76 - 5 \times 10^{-5}$ cm. Hg. It is calcd. that the smallest detectable leak would correspond to a current variation of 4 microamperes and show a pressure increase of 4×10^{-6} mm. Hg/sec. in a one-liter vessel.

Romand, J., V. Schwetzoff, and B. Vodar. "Some Considerations on the Detection of Gases and the Measurement of Their Pressure by Light Absorption in the Far Ultraviolet," J. Phys. Radium, 12, 633-4, May, 1951.

The method is described and examples of its practical applications given; e.g., detection of leaks in apparatus and measurement of low pressures.

Samuel, A. J. "Vacuum Probe, Standard Leaks, and Needle Valve for Use with the Helium Leak Detector," D-4,440.1, Tenn. Eastman Corp., Aug. 16, 1945.

Part I of this report deals with the development and use of a vacuum probe for leak hunting with the Helium Leak Detector. Part II describes two types of Standard Leaks, one adjustable, the other fixed, which were designed for measuring the sensitivity of the Helium Leak Detector by the two leak method. Both types have been proved satisfactory in laboratory and plant tests. Part III deals with the development of a needle valve with fine adjustment for controlling the flow of a standard mixture of helium and air into the leak detector for a sensitivity check by the single leak method. Part IV describes a worm and gear adjustment developed for use with the 3/8" Kerotest angle valve to facilitate throttling of the Helium Leak Detector on a vacuum header equipped with this valve.

Sibley, C. B., and J. R. Roehrig. "Wide-Range Vacuum Gage," Electronics, 26, 176-7, Nov., 1953.

Covers range 10^{-4}-10^{3} mm Hg in six linear ranges and is an extension of the Downing gauge previously described (G. L. Mellar, Electronics, 19, 142-6, Apr., 1946). The output signal is produced by the collection of ions formed by a constant flux of χ-particles from a radium source. Two collecting ionizing chambers are used depending on range, the ion current amplified by a factor of 10^{7} and indicated on meter and recorder. Contamination of electrodes is negligible as low-voltage collecting field is used. Non-linearity limiting the use of previous gauges has been excluded without sacrificing inherent advantages. Data are given on the sensing element, the Roberts d.c. amplifier and the complete circuit. Complete sensing element including preamplifier is mounted in a shell similar to that of a metal 6L6.

Spalding, D. B. "Simple Manometer for Use in Measuring Four Air Velocities," J. Sci. Instrum., 27, 310, 1950.

Describes the construction of two simple instruments for measuring steady differential air pressure down to 0.002 mm of water column.

Spalding, T. R. "Leak Detector for Pipe Joint," U. S. Atomic Energy Commission, U. S. Patent 2,759,175, Aug. 14, 1956.

A leak detector was developed for the joints of a pipe carrying a liquid, the loss of which is undesirable either because of its value or its toxic effect on personnel. A perforated layer of electrically insulating material is secured about the joint, then an electrically conducting foil, and finally a moisture proof outer covering to prevent erroneous signals due to condensation. An electric potential is impressed between the foil layer and the pipe resulting in an open circuit that may be closed only by leakage from the pipe. Any current flow between the pipe and the foil will close a relay and sound an alarm.

Spencer, N. W., and R. L. Boggess. "On the Use of Ionization-Gage Devices at Very High Altitudes," Contr. AF 19 (604) 1890, Mich. U. Res. Inst., Ann Arbor, Jan., 1958.

The use of an ionization gage - omegatron combination in rockets and satellites at very high altitudes is considered. Appropriate instrumentation employing these devices is believed to offer good possibilities for the measurement of atmospheric pressure, temperature, density, and composition.

Storruste, A. "A Simple Mass Spectrometer for Leak Hunting," K. Norske Videnskab Selskabs Forh., 22, 6, 18-20, 1949.

A small (50 by 38 mm) mass spectrometer for leakage hunting is described, in which hydrogen is used instead of the more expensive helium, and in which the radius of the ion path is only 12 mm. There is no separate evacuating system. Both the ion source and the collector box are in the magnetic field of 1350 gauss. In the pressure range 10^{-5} to 10^{-3} mm Hg, the maxima for H^1, H^2, and H^3 correspond to accelerating voltages 100, 50, and 35 v, respectively. Either a d-c amplifier with a meter or an oscillograph are used for the measurement of the output current.

Stott, F. D. "Sonic Gas Analyzer for Measurement of CO_2 in Expired Air," Rev. Sci. Instrum., 28, 11, 914-15, Nov., 1957.

A sonic gas analyzer intended primarily for estimation of CO_2 in the physiological range of 0-10% is described. The instrument uses a resonant cavity maintained in oscillation by a transistor amplifier

as the measuring device; the change in frequency caused by the presence of CO_2 in the cavity is linearly related to the amount of CO_2 present. As described the instrument is intended for continuing sampling at a rate of about 2 litres/min. The time of response is about 0.5 sec for full deflection.

Thomas, H. A. "Development of a Cold Cathode Ion Source for a Mass Spectrometer Type Vacuum Leak Detector," Bull. Agr. Mech. Coll. Tex., Expt. Sta. Bull. 101, 1-58, 1944.

Various design features, together with a discussion of performance. 35 references.

Turner, R. P. "Fluorescent Penetrats with Black Light," Welding Journ., 37, 1167-71, 1957.

Detector is black light. A dot glows at site of leak. It has fair sensitivity. The experiment is very easy and is a manual procedure and therefore slow. This method is used as an in-process check for leaks and is not used as a final check. A person sprays this fluorescent material wherever a leak is suspected and checks it with a black light. If a leak is present there will be a glowing spot when examined. This, like the halide detectors, would be time-consuming for large areas.

Ubisch, H. von. "An Investigation on Hot-Wire Vacuum Gauges," Ark. Mat. Astr. Fys., 34A, Paper 14, 1-33, 1947.

Filaments of Mo, Ni, W and Pt, of various cross-sections (measured microscopically) and lengths, maintained at various temperatures in air. H_2 and CO_2 were investigated. Some filaments were in narrow glass capillaries or in spiral form. Identical filaments formed opposite arms of a Wheatstone bridge, of which the other two arms were precision resistors. With a manually operated balanced bridge, pressure changes of 10^{-5} mm Hg could be detected at low pressures, the useful range being extended to about 30 mm Hg pressure for thin filaments. With a direct-reading bridge with constant current supply, the sensitivity and calibration curve are influenced markedly by the galvanometer current. A theory of the distribution of temperature on a filament is developed.

Varadi, P. F., L. G. Sebestyen, and E. Rieger. "Spectromètre de Masse H. F. et Son Utilisation Dans la Technique Du Vide," Vakuum-Tech., 7, 13-16, 1958.

A portable high-frequency mass spectrometer is described. A simple spectrometer with low resolution power was designed for the investigation of leaks. It operates in the range from 10^{-3} to 10^{-7} Torr and can detect leaks up to 2.8×10^{-8} Torr/sec. The installation can be used also as an ionization manometer for the measurement of the total pressure.

Walter, J. E., and W. D. Hershberger. "Absorption of Microwaves by Gases, II," J. Appl. Phys., 17, 814-22, Oct., 1946.

The absorption coefficients and permittivities of 16 gases have been measured at the two wavelengths $\lambda = 1.24$ cm and $\lambda = 3.18$ cm. The gases are H_2S, COS (CH_4) O, C_2H_4O, C_2H_5Ci, SO_2, NH_3, 6 halogented methanes and 3 amines. Improvements in technique are described, which permit detection of absorption coefficients as small as 0.2×10^{-4} cm^{-1} and measurement of large coefficients with an accuracy of $\pm 5\%$. The measured permittivities at these wave lengths are essentially equal to the static values. A quantitative interpretation of the absorption coefficients in terms of the known structure and spectra of the individual molecules is given. The theory indicates that all non-planar molecules which possess a permanent dipole moment should show appreciable absorption in the microwave region.

Walker, R. E., and A. A. Westenberg. "Precision Thermal-Conductivity Gas Analyzer Using Thermistors," Rev. Sci. Instrum., 28, 10, 789-92, Oct., 1957.

The characteristics of a thermal conductivity gas-analyzer unit employing thermistors as sensing elements are analyzed theoretically. It is shown that, as a consequence of the fact that thermistors have a negative temperature coefficient of resistance, the sensitivity of the instrument as a function of bridge current exhibits a maximum. The dependence of zero stability on current and cell temperature is approximately related to differences in resistance, temperature coefficient of resistance, and geometry of the reference and sample thermistors. An apparatus suitable for precise trace analyses is described and results obtained with it are presented. It is suggested that the widespread lack of faith in the

inherent stability of thermistors for analysis applications is not justified, and has probably been due to insufficient care with current and temperature control.

Warnecke, R. J., Jr. "The Omegatron and Its Applications," Am. Radioélect., 12, 258-81, July, 1957.

An account of the theory of the Omegatron is given and a simple construction described. The use of the device as a leak-detector, both by the normal and accumulation techniques, is described. The use of argon as a probe gas was studied. Resolving power was improved by using fields up to 5000 Oe. A description of an all-metal version of the tube is included.

White, W. C. "Leak Detector Based on a New Principle," Le Vide, 4, 20, 584, 1949.

Description of the General Electric leak detector with Freon gas.

White, W. C. "New Electric Device to Detect Leaks of Inflammable Gases," Elec. Engr., 73, 129-37, 806, Sept., 1954.

Electronic device is similar to halogen. Detector is a ceramic core, approximately the size of a paper clip, made at Alumina with a small amount of an alkali metal salt present. Natural gas, alcohol vapors, water vapor, hydrogen, and other gases are used. From pressurized system to atmosphere. Is used with same electronic system as halogen detector. Only approximate: Hydrogen - 1 in.^{3}/hr. Ease and speed probably same as with halogen detector. A ceramic rod approximately the length of a paper clip is made of alumina with a small amount of a alkali metal salt added. The rod is heated by a platinum wire wound around it. The platinum wire is one electrode and the rod itself is the other. The resistivity of the platinum wire-ceramic junction is highly sensitive to small amounts of inflammable gas in the air. Probably the device should be made more sensitive and could be calibrated.

White, W. C. "Positive-Ion Emission, a Neglected Phenomena," Inst. Radio Eng. Proc., 38, 852-8, Aug., 1950.

Distinction between ordinary electron emission, upon which common tubes depend, and positive ion emission which can be used in electrode devices operating in air; details of leak detector utilizing phenomena of positive ion emission; further possibilities of positive ion devices.

Young, Allan C., and W. A. Robinson. "Small Sample Volume Infra-Red Carbon Dioxide Analyzer," Report on Contr. AF 33 (038) 422, 1-10, Dec., 1954.

A compact CO_2 analyzer using an interrupted infra-red beam is described. The sample chamber has a volume of 1 cc. Ninety per cent response requires a time of 1/25 sec and a volume flow of 1 cc.

No Authors:

"Refrigeration Chemical Helps Electronic Nose Find Leaks," Elec. Eng., 72, 471, May, 1953.

Electronic detection of chemical. Freon-12 afluorinated hydrocarbon. Long distance telephone cables, distillation tanks the size of two-story building. From pressure to vacuum. Can detect 1/100 oz of Freon per year. Electronic device weighing 17 lb and ~size of portable typewriter. Relatively easy. Huge distillation tanks as large as two-story building are tested by filling with Freon and then checking likely spots - joints, fittings, etc. - with the portable electronic tester. Western Electric has developed a detector to ride along long distance cables (like a monorail) and check for leaks. Checks 120 feet of cable per minute. No information is given as to how the electronic detector works, but probably could be obtained from G.E. who developed it.

"Alarm Devices for Indicating the Presence of Dangerous Gases and Vapors," Chemisch-Technische Reichsanstalt., Z. ges. Schiess-u. Sprengstoffw., 34, 337-9, 1939.

Upper and lower explosive limits are given for 18 combustible gases in mixtures with air, and dangerous lethal conens.are given for 16 poisonous gases. The requirements which a satisfactory alarm device should meet are listed. All com. devices tested by the Chemisch-technische Reichsanstalt and by certain other testing stations are named and the principle upon which they operate are described briefly.

"Detecting Fuel Leaks with 'Black Light'," Diesel Power, 82, 10, 48-9, Oct., 1954.

Use of Magnaflux ZB 26 Black Light on Southern Railway for finding locomotive fuel line leaks; device in high voltage mercury are light, equipped with fluted violet filter, which brings out natural fluorescence of diesel fuel oil as contrasted with that of lubricating oil.

"Demonstration of Aqua-Visual Apparatus for Leak Detection at Sheffield, Water & Water Eng., 57, 687, 197-9, May, 1953.

Leak detector consisting of microphones and amplifiers, records sounds produced by underground leaks; tests and results are described.

"Mass Spectroscopy in Physics Research," U.S. Bur. Stand., Circular 522, 1-278, Jan. 23, 1953.

Symposium papers include:
J. Mattauch: Double-Focusing Mass Spectrometer.
A.O.C. Nier: Accuracy of Mass Spectrographic Isotope Mass Measurements.
H. E. Wald: Discussion of Masses Derived from Recent Doublet and Q Measurements.
K. T. Bainbridge: Use of Mass Spectrography in Study of Nuclear Shell Structure.
H. E. Duckworth: On Substandards of Atomic Mass.
K. Ogata and H. Matsuda: Masses of Hydrogen, Carbon, and Some Nuclides of Medium Mass.
T. L. Collins: New Doublet Measurements.
J. Mattauch and R. Bieri: Some Recent Investigations in Electron and Ion Optics.
N. Svartholm: Increase of Sensitivity of Mass Spectrographs by Concentration of all Beams in One Point, Producing Special Line.
R. F. K. Herzog: Improved Focusing in Combinations of Radial Electric and Magnetic Fields.
H. Hintenberger: High Resolution in Conventional 180° Mass Spectrometers.
C. E. Berry: Influence of Stray Field on Focusing Qualities of Magnetic Sector Field.
W. Paul: Nonmagnetic Radio-Frequency Mass Spectrometer.
W. H. Bennett: Time-of-Flight Mass Spectrometer.
E. E. Hays, P. I. Richards, S. A. Goudsmit: Mass Synchrometer.
L. G. Smith: Work on Cyclotron Resonance at National Bureau of Standards.
J. A. Hipple and H. Sommer: Present Status of Isotopic Abundance.
A. O. C. Nier: Mass Spectrometer for Measurement of Small Differences in Isotone Abundance Ratios.
S. Epstein: Instrumental Problems Encountered in Mass Spectrometer Isotope Analysis of Water Samples.
H. W. Washburn, C. E. Berry and L. G. Hall: Mass Spectrometer

as Tool for Studying Nuclear Reactions.
M. G. Inghram: Electromagnetic Separation of Noble Gas Isotopes and Their Use in Some Nuclear and Spectroscopic Experiments.
J. Koch: High-Intensity Electromagnetic Isotone Separator.
J. Kistemaker and C. J. Zilverschool: Measurement of Trace Quantities of Uranium and Lead in Minerals and Meteorites.
D. C. Hess, H. Brown, M. G. Inghram, C. Patterson, and G. Tilton: Further Results on Double Beta Decay of Te^{139}.
R. J. Hayden and M. Ginghram: Ionization by Electron Impact.
H. D. Hagstrum: Mass Spectrometer Effects at Higher Pressures.
F. J. Norton: Observation of Ion Dissociation in Parabola Spectrograph.
A. Henglein and H. Ewald: Direct Measurement of Appearance Potential and Ionization Probability using Mass Spectrometer.
R. E. Fox, W. M. Hickam, T. Kjeldaas, Jr., D. J. Grove: Mass Spectra of Heavy Hydrocarbons.
M. J. O'Neal: Space Charge and Error in Mass Spectrometric Measurement.
E. W. Becker and W. Walcher: Mass Spectrometric Studies in Solids.
T. H. Plumlee: Distribution of S^{34} in Nature.
H. G. Thode and J. Macnamara: Influence of Fractionizing and Viscosity Effects in Mass Spectrometric Gas Handling Systems.
J. Kistemaker: Mass Spectral Patterns of Isotopic Molecules.
O. Schaeffer: Session on Experimental Methods.

"Report," Iron and Steel Eng., 33, 162, April, 1956.

At Phil. Gas Works they deal with combustible gases and this is the method they use to detect them when leaking. They have stationed around the room, at four points, instruments containing two heated, sensitized platinum filaments. One had the atmosphere of the room drawn over it and the other has a "compensator" sealed in a cell containing uncontaminated air. Any combustibles present in the air sample drawn over the first - the detector - will burn as soon as they come in contact with the heated wire. This increases the temperature of the filament and, consequently, its electrical resistance. The higher resistance of one filament upset the balanced electrical circuit in direct proportion to the concentration of combustibles in the sample. They have a warning system when unbalance reaches a preset mark.

"PE Tube Gas Detection," Electronic Industries, 3, 3, 108-9, Mar., 1944.

Illustrations and operating details of ultraviolet photometer designed to analyze for dangerous concentration of harmful gases; device consists of mercury arc ultraviolet generator and quartz condensing lenses and prisms; two large gas tubes connect to analyzing chamber, which contains balanced phototubes and amplifier.

Advertisement of Sonic Leak Detector, Delcon Corporation, Palo Alto, Calif., Industrial Design, 9, 3, 94, March, 1962.

Detects leak in pressure and malfunctions friction joints by listening to production equipment. Translates inaudible sound of frequencies from 35 Kc to 45 Kc into the audible frequency range. Does not pick up ambient audible noise of any kind. It can operate (with earphones) in the deafening din of a tool shop or near a whining jet engine. Works on flash light or mercury cell batteries. Weight is 8 pound.

CHAPTER XII

CONCLUSIONS AND RECOMMENDATIONS

Many methods and many instruments are available for the detection and location of leaks. No one instrument or method is ideal. Some methods are more sensitive than others, some are more convenient. The most sensitive known methods require the use of tracers and are more or less subject to noise and contamination problems. Sonic methods in their present state of development have the convenience of not requiring tracers and thereby avoiding contamination problems, but lack sensitivity.

In the face of these difficulties and imperfections there is still much that can be accomplished by optimization of the technique available.

The sensitivity of the halogen leak detector is such, for example, that with proper use it can detect very small leaks. But this inherent sensitivity cannot be reached in practice if there is background contamination. A large leak near a small one may completely swamp the signal from the small one. If, for example, one were using 1000 PPM tracer and sniffing a leak of 0.007 SCIM he would expect a reading of, say, 1 on the dial with the H sensitivity setting, but a nearby large leak could provide enough contamination to show off-scale reading even on the intermediate 0.1 sensitivity setting. This, of

course, completely obscures the smaller leak.

The gradient detector - essentially two halogen detectors in a bridge circuit - shows considerable promise of a solution to this problem.

The low diffusion coefficient of the heavier gases such as Freon, gives rise to another problem which must be overcome in making best use of the halogen detector. In order to produce a dependable signal at leaks, the tracer gas mixture with which the system is charged must be uniform in composition. Any blind passages in the system must be flushed with well mixed tracer gas - otherwise leaks in these cul-de-sacs will simply leak air and escape detection. Those not flushed will remain at low halogen concentration for long periods because of the low diffusion rate of Freon 12 in air (about 0.3 moles/ft^2 hr per unit molar concentration gradient). The time required for a blind duct one yard long to reach 50% of full halogen concentration is on the order of 3 hours if diffusion alone is acting. Fig. 77 shows the time required for various lengths of blind duct to reach 10% and 50% of open end halogen concentration.

On the other hand, settling out of heavier Freon from an initially well mixed tracer gas is not significant. The equilibrium distribution law gives the ratio of concentration at top of a 100 foot tank relative to that at the bottom as:

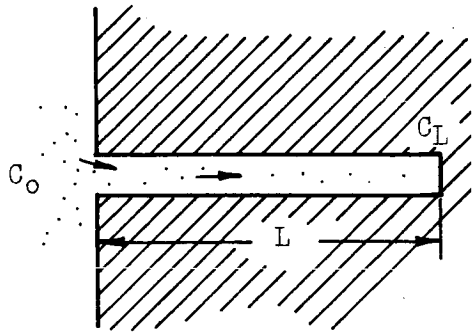
$$T \simeq \frac{L^2}{\pi^2 K} \ln \frac{4}{\pi} \frac{1}{1-C_L/C_o}$$

K = F-12 Diffusion Coeff $\simeq$ 0.3 Ft^2 /Hr

C_o = F-12 Concentration in Tracer

C_L = F-12 Concentration at End of Duct

T = Time - Hours



(Pressure = 1 Atmosphere)

NOTE - For Pressures Greater Than 1 ATM,
Multiply Time Shown on Graph
by Pressure in Atmospheres.

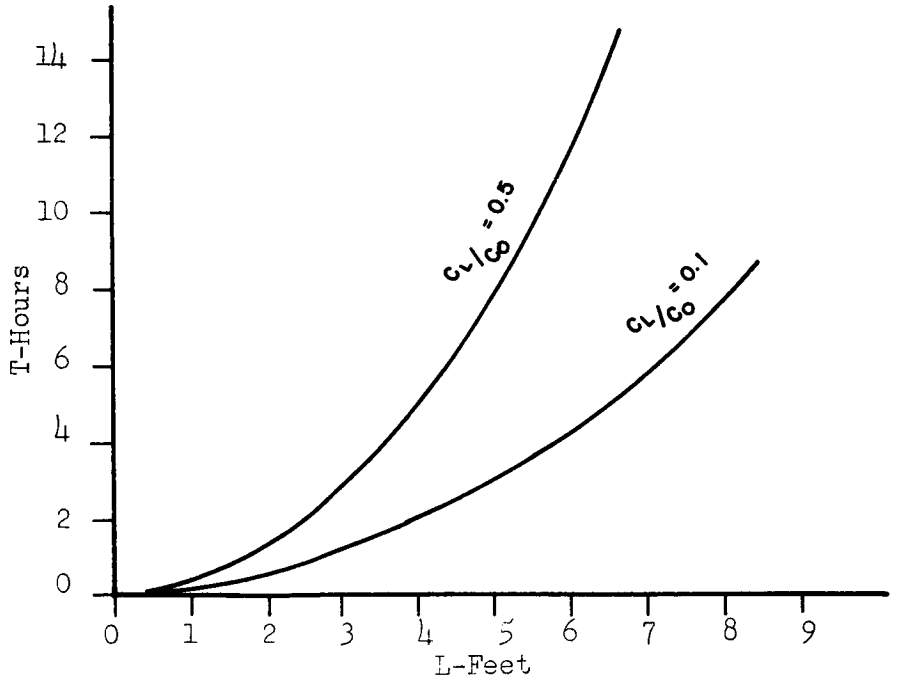


FIGURE 77

DIFFUSSION TIME VS LENGTH OF BLIND DUCT

$$C_{100}/C_o = \exp(-MWh/RT) = 0.98$$

KW = molecular weight = 121 lb/lb-mole
h = height of tank in feet = 100 ft.
R = universal gas constant = 1545
T = temperature = 535° R.

There should, therefore, be no pooling problem inside the system provided precautions are taken to mix the tracer thoroughly with air used in pressurization of the system.

A technique for charging the system with thoroughly mixed gas of correct concentration is shown in Fig. 78. Freon is bled into the system through a rake, mixed in 50 or more diameters and then sampled through another rake and tested. The dial reading and Fig. 79 can be used to verify the desired concentration and leak detection capability of the tracer gas mixture. For example, if it is desired to detect leaks of 0.004 SCIM, a meter reading in the sample gas should be about 3 on the 0.01 sensitivity setting, and the flow of Freon should be adjusted to give that reading on the G. E. meter.

It should be noted, however, that if the system is not being purged with tracer gas before pressurization starts, but tracer is simply being added to air already in the system, uniform mixture will not be so readily achieved. In any event, even after the pressurized system has come to an equilibrium mixture, the equilibrium concentration will be less than the concentration of the admitted tracer gas by a factor of (P-1)/P where P is the final pressure in

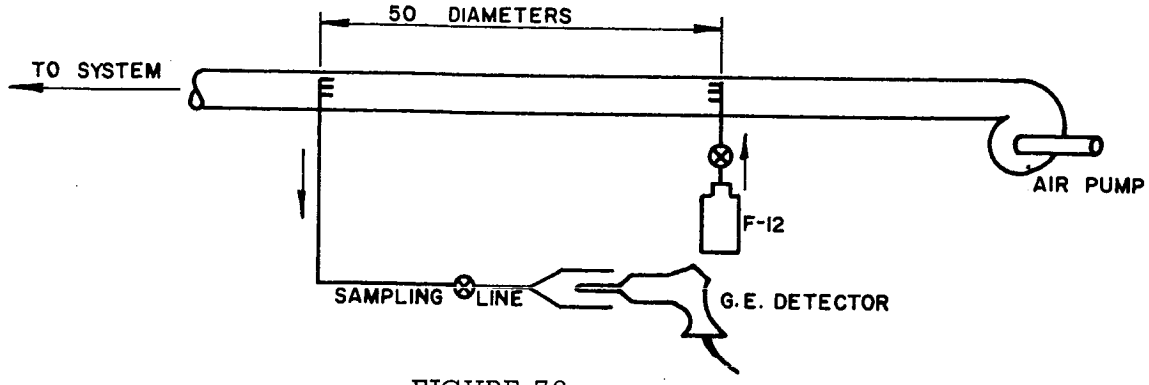


FIGURE 78

TRACER MIXING AND SAMPLING ARRANGEMENT

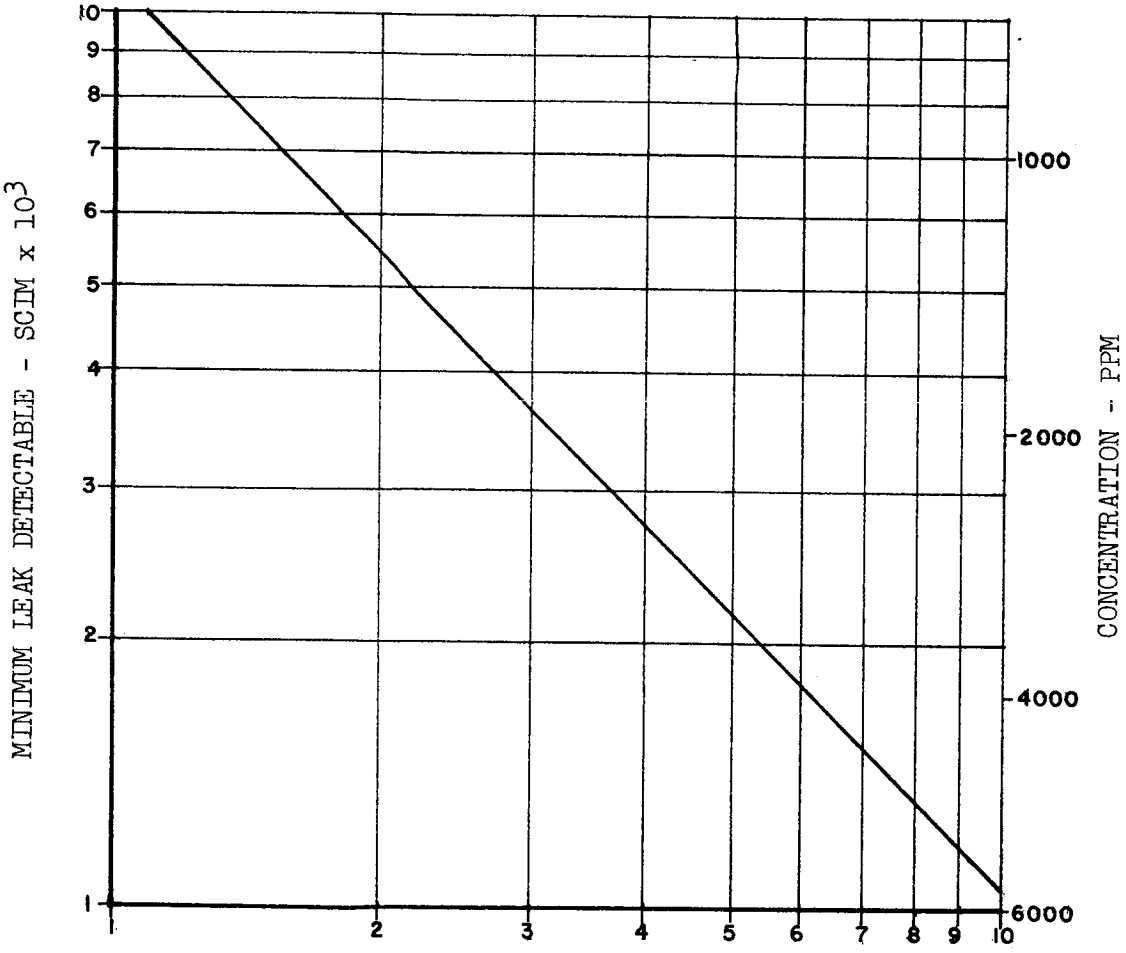


FIGURE 79

DESIRED CAPABILITY VS GE METER READING IN TRACER

atmospheres. For example, if a system is pressurized to 1.5 atmospheres by a tracer gas of 1000 PPM, capable of detecting a leak of 0.007 SCIM, the resulting concentration in the system after the air and tracer have come to equilibrium will be only 1000 x (1.5-1)/1.5 or 333 PPM and the resulting minimum detectable leak will be increased by the inverse ratio to 0.021 SCIM.

If it is desired to use a tracer gas of concentration above 6000 PPM where the meter reading in tracer gas would be off scale, the simple dilution scheme shown in Fig. 80 may be used.

For instance, if leaks of 0.0005 SCIM are to be detected, the tracer should be of such concentration that a 1/10 dilution of it would read on the meter about 2 (same as full strength tracer for 0.005 SCIM leak). This dilution may be obtained by adding 10 ml of tracer to the 100 ml graduate and then adding air through the 3-way valve to make 100 ml total volume. This sample can then be ejected over the detector and the reading noted.

If continuous sampling is preferred, the arrangement shown in Fig. 81 may be used, where the two flow meters are to be adjusted to show the desired proportion of air dilution.

Note, rubber and plastic tubing should be avoided in any of these arrangements since Freon is easily absorbed by these materials and may be released later to show false concentration readings.

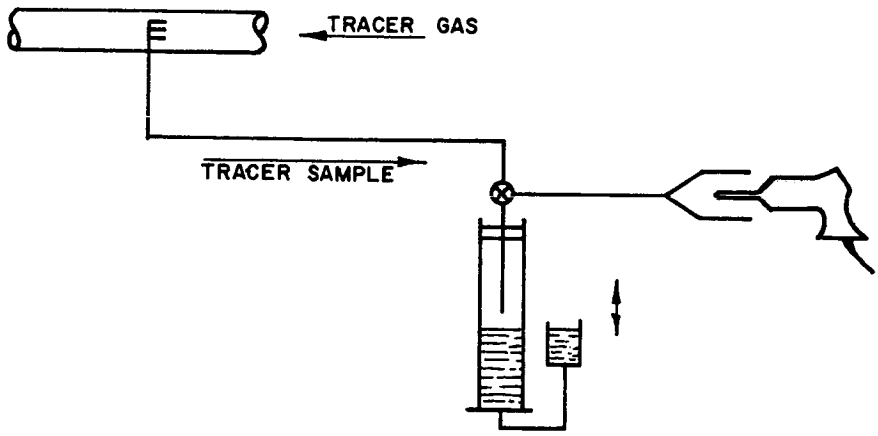


FIGURE 80

TRACER DILUTION SCHEME

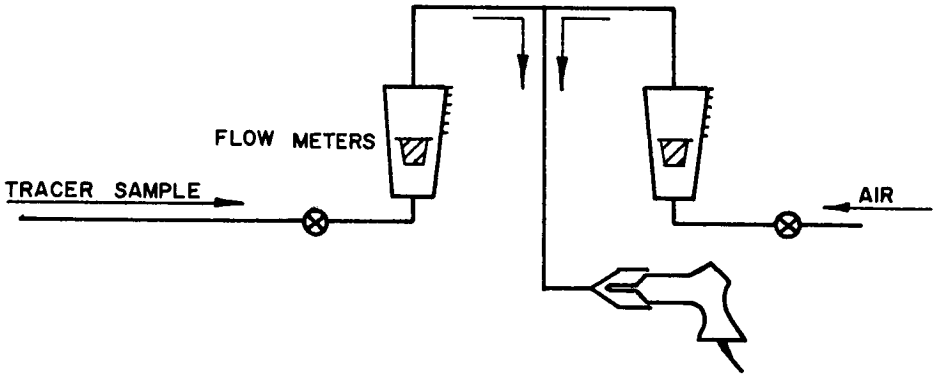


FIGURE 81

CONTINUOUS SAMPLING SCHEME

In order to make optimum use of the capabilities of the halogen leak detector in either its simple concentration sensing form or in some more sophisticated modification such as the halogen gradient detector, the following procedure seems advisable:

A. Check for larger leaks by using the Delcon Ultrasonic Translator or similar ultrasonic detector. This step should be time saving and should permit stopping of the larger leaks before excessive contamination of the ambient has occurred.

B. Charging the system with Freon tracer should be the last step in leak checking, after larger leaks have been corrected and other tests made.

C. Freon tracer should be pre-mixed. Its concentration should be checked by appropriate metering to avoid over- or under-rich mixtures.

D. In order to realize the theoretical sensitivity of the halogen detector, every effort must be made to reduce halogen concentration in the atmosphere around the test area. All dumped tracer gas should be ducted away from the test area, outside the building, or possibly back to the compressor for storage in a tracer tank. Even with a gradient detector this procedure vastly improves location efficiency. Background contamination cannot be compensated for by increasing Freon concentration in the tracer since the background is itself proportional

to this concentration.

E. During charging with tracer, deadend ducts should either be opened to allow trapped air to escape or time should be allowed for diffusion of Freon to take place (this diffusion time can be quite long, cf. Fig. 77).

With these precautions, the halogen detector or the halogen gradient detector will perform satisfactorily on even very small leaks.

Suggestions for Future Investigations

Since the effective development of any field must rest on a firm foundation of knowledge of the basic processes involved, it seems of first importance to suggest that the study of leak phenomena be made a continuing part of any program for the improvement of leak detection techniques. A better knowledge of concentration and gradient patterns around leaks is essential to systematic studies. The approximations now in use are based on simplifications and idealizations many of which are known to be seldom applicable.

One line of investigation which could provide very useful information involves suggested methods of obtaining concentration fields by means of a liquid analog of the gas leak. This is feasible since the process of diffusion in liquids obeys the same laws as does gaseous diffusion.

The method would use a low melting solid just above its melting

point. The geometry of interest would be provided and a dye permitted to diffuse into the liquid. After a calculated lapse of time, the liquid would be allowed to solidify in such a manner as to create a minimum of disturbance in the system. A careful selection of the media involved would result in a frozen diffusion pattern which could then be investigated by slicing the whole along parallel planes which would yield concentration patterns by photography or measurement (comparison with samples of known concentration, for instance).

A simpler process which yields information concerning concentrations in the plane of the leak provided this is a plane of symmetry has been investigated. This involves simulated geometry and a metol or hydroquinone tracer in a water filled system. These chemicals are allowed to diffuse into the ambient water through the leak and, by absorption, charge a piece of exposed photographic film or paper with developing agent proportional to the concentration in the plane of the paper (plane of the leak). The pattern may then be developed by treating the paper or film with alkali.

It is felt that these processes are worthy of investigation as tools of basic research in gas dynamics.

The energetics of leak dynamics is another field ripe for basic studies which may well lead to significant breakthroughs in the improvement of leak location techniques. The injection of various forms

of energy into the system and its detection through leaks has fascinating aspects, some of which seem to be about to pay off. Sonic injection has been mentioned as one of these. Already this has shown fantastic sensitivity in the location of underground gas leaks at low pressures. There remains a whole field of communications techniques to be applied in the refinement of this process.

It is far from inconceivable that acoustic methods, either active or passive or both, may find use in space environments. Here, in low noise ambients, the high sensitivity and extremely small size of newly discovered semiconductor microphones may make it possible to automate a leak detecting, measuring, and remote reporting system. Such a system might employ a pattern of small transducers to feed phase and amplitude information into a miniaturized special purpose analog and/or digital computer which could calculate the location of the leak and its magnitude and while reporting also take elementary action to correct the situation.

It should be emphasized that it is not wise to discount currently unusable methods and instruments. Many of these may await only some small improvement to make them highly competitive with the best presently available techniques. Molecular electronics is fast becoming state-of-the-art. This means that much more sophisticated circuitry can be used than has been possible up to the present.

Servo balancing and servo adjusting techniques may be applied to such instruments as the thermistor bridge to permit saturation effects. Time sharing of a single diode in the halogen gradient detector has attractive features as does the redesign of the diode to provide a split cathode for bridge use.

A wisely planned program should push basic studies and be ever alert for new developments of promise.

APPENDIX A-1

GENERAL ELECTRIC HALOGEN LEAK DETECTOR H-2

Operating Safety Precautions

WARNING

Do not use the Leak Detector in any combustible or explosive atmosphere. The leak detector element operates at a temperature in the range of 1300 F (700 C) to over 1600 F (900 C).

The Control Unit, Cat. No. 5470201G1, is designed to be used in conjunction with halogen-type leak detectors. Due to the high operating temperature of the leak detector element, and the high voltages in the amplifier circuit, the following operating safety precautions must be observed:

(1) Never enter or test in an area where there is an explosive vapor. If there is a question, first test the area with an explosion meter.* (The lower explosive limit of hydrogen in air is approximately 4 percent.)

(2) Never test in vents to enclosed spaces, such as bearing housings, oil tanks, or piping, etc., without first testing with an explosion meter.

(3) Since voltages as high as 300 volts are present in the amplifier circuit, the case should be kept at ground potential by using a

three-prong grounded receptacle or the adapter plug supplied. If the adapter plug is used, make certain that the ground lead is connected to a good "earth" ground.

Preparation for Use

(1) Place Control Unit in the desired location. Raise the cover, then unwind enough cable to reach the power outlet.

(2) The unit is shipped connected for 110-127 volt, 50-60 ops operation. (The Control Unit can be used with a 220-254 volt, 50-60 cps source, provided that the proper internal connections are made and the fuses are changed.) If a receptacle to fit the three-pronged plug is not available, use the adapter supplied, making sure that the ground lead is connected to a good "earth" ground.

(3) Connect the Leak Detector to the Control Unit, following the instructions supplied with the detector.

Operational Use

(1) To operate the Control Unit and Leak Detector, proceed as follows:

(a) Turn power on. Rotate VOLUME knob clockwise until a click is heard or felt. Further clockwise rotation will increase the volume of the audible alarm.

(b) If a rattling sound is heard, turn the air-pump adjusting screw counter-clockwise until the rattling stops. (This screw is

reached through a hole in the bottom of the cable compartment.)

(2) Check air flow:

(a) Turn both three position switches on the Control Unit to the AIR FLOW position.

(b) Indicator should read approximately "0". If reading is not approximately "0", reset using the AIR FLOW ZERO ADJUSTMENT.

(c) Lift the CHECK AIR FLOW cap (upper right-hand corner of control panel) and insert the Leak Detector probe. If the indicator is not approximately at the point marked AIR, correct it, using the air-pump adjusting screw.

Note - If the indicator readings specified above can not be obtained, further adjustments are required as described in the Air Flow Calibration immediately below.

Air Flow Calibration

(1) Check the air pump for optimum adjustment by first turning the adjusting screw (in the cable compartment) clockwise until the pump rattles, then backing off one-half of one turn.

(2) If this does not produce the correct flow, check the air flow at the detector connector in the following manner:

(a) Unplug the detector unit.

(b) Readjust pump as described in Step 1 above.

(c) Attach short piece of tubing to the air connection on the

pump. Set control unit to the AIR FLOW position, and insert the other end of the tubing into the CHECK AIR FLOW fitting, (or in an air-flow measuring instrument, Cat. No. 5469975G1).

(d) The Control Unit indicating instrument should read over 8. (An air-flow meter should read over 2 scfh.) If the pump and connections are in good condition, check the detector-unit air system according to the applicable Leak Detector instruction book.

(3) If both the pump and the detecting means appear to be operating properly, and a correct air-flow reading still cannot be obtained, the air flow meter may be at fault and should be checked as follows:

(a) Check the calibration of the air-flow meter by placing a Cat. No. 5469975G1 air-flow measuring instrument (available as an accessory) in series with the meter and leak detector. With both three-position switches in the AIR FLOW position, turn the AIR PUMP control until the air-flow measuring instrument, Cat. No. 5469975G1, indicates a flow of 1.0 to 1.2 scfh. The Control-Unit indicating instrument reading should be within the AIR range. If it is not, adjust it using the CALIBRATE AIR FLOW

control. (Remove the knurled cap to adjust control.) Be sure the instrument is adjusted to zero before taking a reading. If the meter cannot be zeroed and/or calibrated, the air-flow sensing element may be defective, and should be replaced.

(3) Check the power to the heater of the sensitive element:

(a) Set the right-hand three-position switch on the Control Unit to the HEATER position.

(b) Adjust the heater power, by turning the knob near the loudspeaker inside the Control Unit case, as follows:

When using the Control Unit with a Type H-2, -3, or -5 Leak Detector, turn the knob until the indicator pointer falls within the band marked HEATER.

When using the Control Unit with a Type H-4 Leak Detector, turn the knob until the pointer falls within the band from 2 to 4 on the indicator scale.

(4) Adjust the loudspeaker cut-in point (or control relay when used):

(a) Set the right-hand three-position switch on the Control Unit to OPERATE.

(b) Set the indicator pointer at the desired cut-in point, by rotating the ZERO SET knob on the Control Unit panel. (A cut-in point in the range from 2 to 3 is recommended.)

(5) Final Adjustments:

(a) Set the right-hand three-position switch to the OPERATE position (if not there already); then set the left-hand three-position switch to select the type of operation desired, as follows:

The MANUAL ZERO position gives continuous response to a leak, and also the highest sensitivity. However, varying background levels will affect zero.

The AUTO ZERO position gives an indication when a leak is discovered, but automatically returns to zero even if the probe is not removed from the leak. Slowly varying background contamination will have little effect on the zero setting.

(b) Select sensitivity desired. HIGH gives the greatest output response. Settings of 0.1 to 0.01 give a corresponding fraction of HIGH sensitivity. When using the Control Unit with a Type H-2 Leak Detector, the SENSITIVITY and ZERO functions can be transferred to the gun by setting the SENSITIVITY control to GUN. If, however, it is preferred to use the controls on the Control Unit rather than those of the gun, set the SENS knob on the gun to the H (high) position.

(c) Set indicator pointer to zero (no leak condition) by rotating the ZERO knob clockwise for upscale movement, counterclockwise for downscale.

APPENDIX A-2

OHIO UNIVERSITY TWIN HALOGEN DETECTOR BRIDGE

Operating Instructions

(1) Connect power plug to 120V AC outlet. Turn power 'ON-OFF' switch to the 'ON' position by putting the toggle switch in the 'UP' position. This switch is located at the lower right side of the front panel. Turn the control '0-130 Volts' to approximately 115 volts. This corresponds to approximately 12 volts on the heaters of the halogen detector units. Allow the equipment to warm up for thirty minutes so the halogen bridge will stabilize.

(2) With the ends of the two detector input tubes close together so they will be sampling the same concentration of atmosphere, adjust the screw driver adjustment on the lower left side of the front panel until the indicator meter reads 'Zero" (center scale). Then with the General Electric Standard Leak set to a standard leak of .5 ounces per year, place one of the sensing tubes in front of the leak and note the magnitude and direction of deflection on the indicator meter on the front panel. If more or much less than a full scale indication is noted, adjust the sensitivity control for a reading of half scale. This control is located to the right of and beside the screw driver adjusted centering control.

Note — The other tube should be held a few inches or more away from the

leak so they both do not sample the same concentrations of halogen. This insures that the bridge will detect any detectable differential of the two samples of gas. Next, use the other tube to sample the leak in exactly the same manner as with the first tube. The indications should be the same within ± 10%. If they are not the same, the control located inside, accessible through the top door, should be adjusted experimentally until the two indications are the same. This control adjusts the heaters of the halogen detectors so a balance in sensitivity of the two detectors is obtained.

(3) The bridge is now ready for use in locating an unknown leak. Advance the sensitivity control to its maximum clockwise position and proceed to probe with the two tubes near the suspected leak. When a leak is detected, a deflection will be obtained. The direction of deflection will be opposite for each of the individual tubes. By noting which direction each individual tube gives, one can determine which tube detects the leak.

APPENDIX A-3

NRL THERMISTOR-TYPE GAS LEAK DETECTOR - MODEL 410

Operating Instructions

(1) Preparation for use

(a) Plug the detector unit cable into the power supply (or battery pack if furnished).

(b) If the electronic power supply is used, plug the 3-prong polarized plug into a 115 volt 60 cycle polarized line receptacle of the same type. (If a polarized receptable is unavailable, use the polarized to unpolarized adapter furnished.)

(2) Checking for normal operation

(a) Switch the power switch to ON and allow the equipment to warm up for approximately 1 minute. (The warm-up process provides a partial check of the equipment. When the power switch is first thrown on, a normally operating unit will give a near full scale meter indication and a bright light indication. As the unit warms up, the brightness of the light will decrease and the meter reading will decay to the normal noise level of 5% of full scale indication or less.)

(b) If the electronic power supply is used, operate the line polarity reversing switch to the position which gives the lowest noise

level. (This switch may or may not have an effect depending upon the nature of the line circuit.)

(c) Make a check of the sensing capability of the detector by holding the sample intake nozzle near the palm of the hand. Sizable indications on both the meter and light should result within approximately 1 or 2 seconds after sampling.

(d) Although not required, a quantitative test may be made by using a known freon F-12 leak from a GE Leak Standard Model LS-10 or equivalent. A meter reading of 25 to 30% of full scale should be obtained from a 3 oz/yr leak rate.

(3) Operational use

(a) Having checked that the detector is operating normally, slowly move the sample intake nozzle along the gas piping, fittings, valves, etc., where leaks may be suspected. (The flexible nozzle may be bent as necessary to reach otherwise inaccessible locations. Extensions to the flexible nozzle or a longer flexible nozzle may be used if necessary, but a longer time lapse between sampling of the leak and detector response must be expected.)

(b) As previously indicated, the design of the gas leak detector is such that it responds only to a change in thermal conductivity of the gas being sampled. As the nozzle is moved past a

sizable leak, the unit will normally give two successive indications as the sampled gas changes from ambient air to the leaking gas and back to ambient air again. If the nozzle is placed at or near the leak, a single indication will result after which the meter will return to the normal noise level. No second indication will be observed until the nozzle is removed from the leak at which time the sample changes from the suspected gas back to ambient air.

Operational Maintenance

Only minor maintenance such as replacement of the indicator lamp and checking of the intake or exit filter screens should be undertaken by operators untrained and unskilled in electronic maintenance and repair.

APPENDIX A-4

G.A.S. HIGH-SENSITIVITY LEAK DETECTOR - MODEL H-4

Operating Instructions

This Gas Analysis Systems instrument may be carried by the hand strap or supported with the neck band. The following five pre-operational steps are suggested:

(1) Attach the pick-up wand to the 'Inlet' connection (near the battery holders). Make sure the connection is hand tight.

(2) If pump suction is to be used, attach the line cord. Pump operation is indicated by a low hum.

(3) Move Switch from 'OFF' to 'A' and position meter needle to 'A' using Calibrate 'A' knob.

(4) Advance Switch to 'B' and position meter needle to 'B' using Calibrate 'B' knob.

(5) With Switch on 'M' (measure), turn 'Gain' knob full clockwise for highest sensitivity. Make sure the pick-up wand is sampling only clean air. Position the meter needle to approximately zero on the meter scale using the 'Zero' knob. Exact zero positioning is not necessary because the Model H-4 is indicating leak intensity and is not measuring quantitatively.

Note - With switch on 'R', the leak intensity signal is reversed on the

meter scale. This permits a direct read-out of the signal generated by almost all gases used in pressurized systems.

When tests are completed, return the Switch to 'OFF'. This setting opens all battery circuits and protects the meter movement.

If it is desired to Hand Pump, the Aspirator Bulb is attached to the 'Outlet' pipe. The instrument should be purged with clean air by pumping 5 or 6 times before starting Step 5. While searching for leaks, a slow rhythmic pumping should be maintained. Purge with clean air to return meter needle to zero after each leak reading.

Maintenance

This G.A.S. High-Sensitivity Leak Detector requires little maintenance except to keep it clean and dry.

(1) When adjustment of either Calibrate 'A' or 'B' knobs will not position meter needle, the batteries should be replaced. These must be inserted in the battery holder with the PLUS (+) terminal IN.

(2) If at any time it becomes impossible to zero the meter with the 'Zero' knob, a supplementary adjustment is available through the 'Black' edged hole near the 'Inlet' connection. Slowly position the blade until the 'Zero' knob regains control of the meter needle.

(3) If the meter shifts off zero when the 'Gain' knob is adjusted, correction may be made as follows. With full gain, zero meter needle with 'Zero' knob (or if necessary readjust through 'Black'

edged hole). Reduce gain to minimum and again zero needle with screw-driver adjustment but through 'Red' edged hole. Repeat these two steps until meter needle does not move with gain change.